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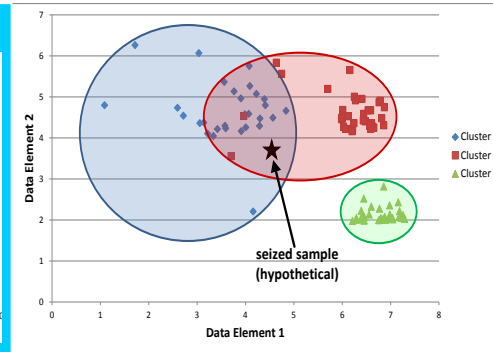
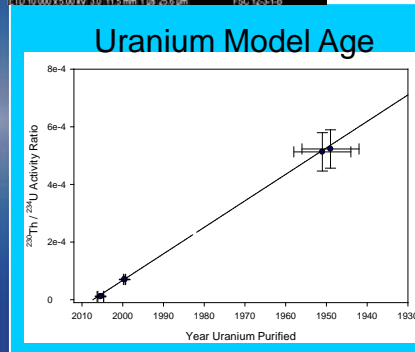
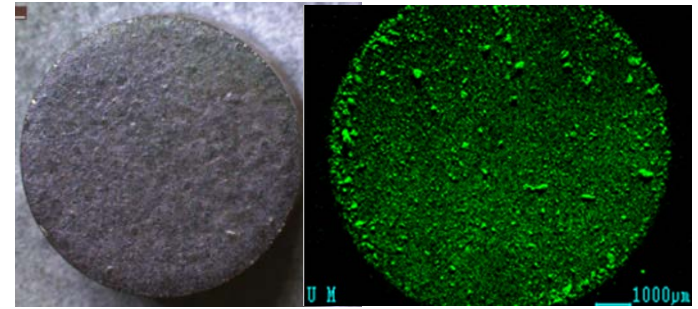
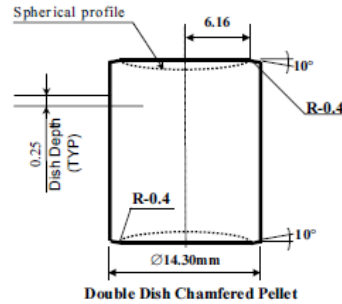
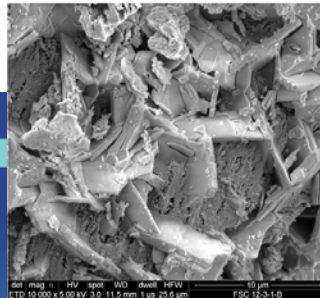
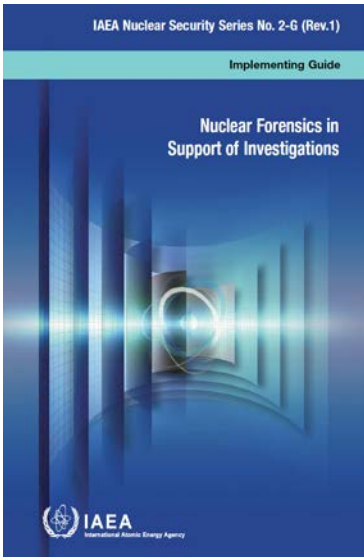
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Nuclear forensics: The fingerprints of mass destruction

Stephen LaMont

Los Alamos National Laboratory

August 2018



Outline

- How pre- and post-detonation nuclear forensics fits into the broader nuclear security regime
- Why LANL?
- Post-detonation forensics and R&D
- Pre-detonation forensics, national nuclear forensics libraries, and R&D
- International outreach and engagement
- Summary

Pre- and post-detonation nuclear forensics

- Nuclear forensics supports the investigation of both illicit activities involving material out of regulatory control (MORC), and incidents of nuclear terrorism

Pre-detonation

- Interdiction / recovery of MORC or device
- RED response
- Investigative leads
- Material provenance

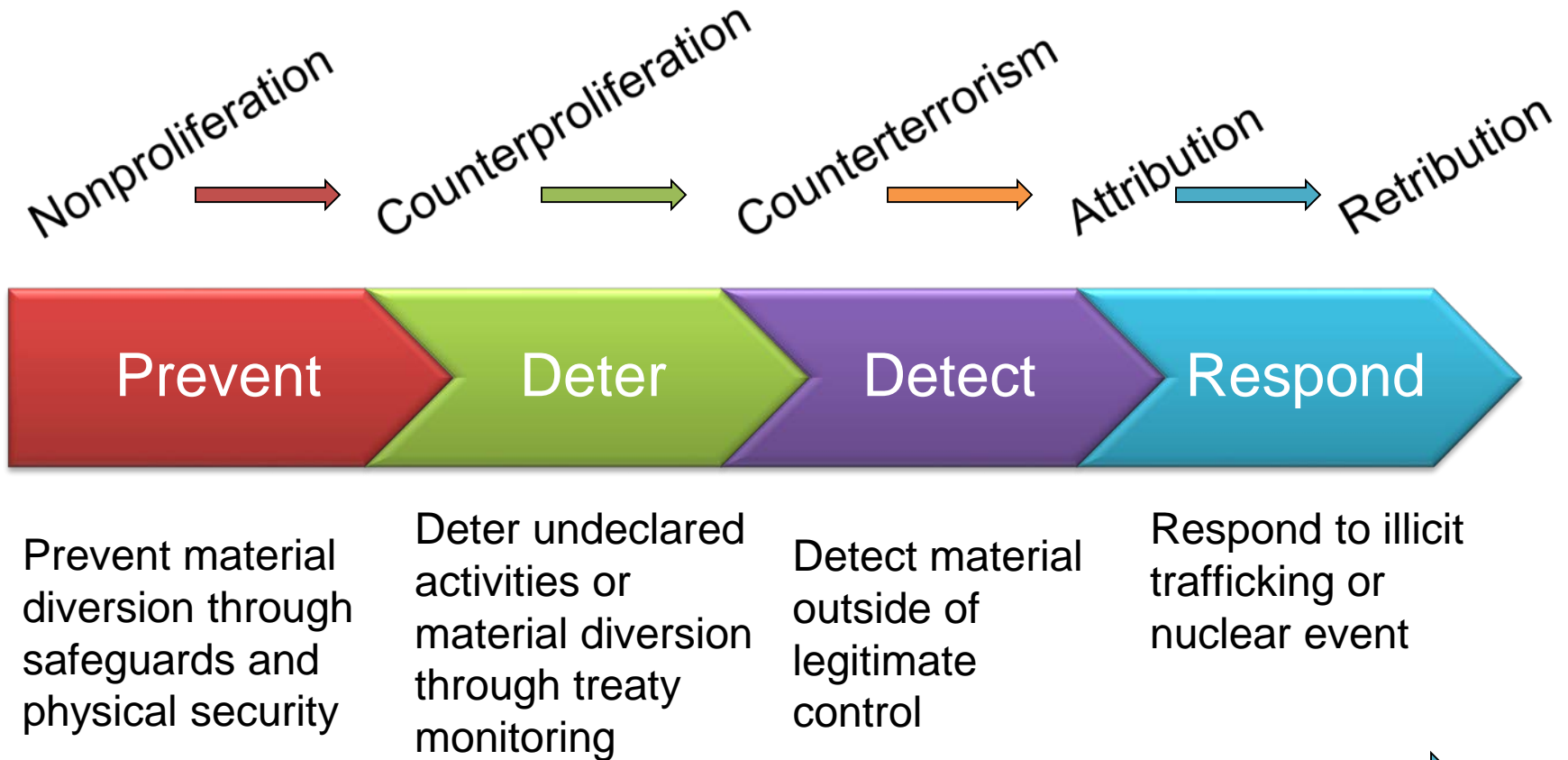


Post-detonation

- Nuclear or RDD explosion
- Event characterization
- Investigative leads
- Device and material provenance

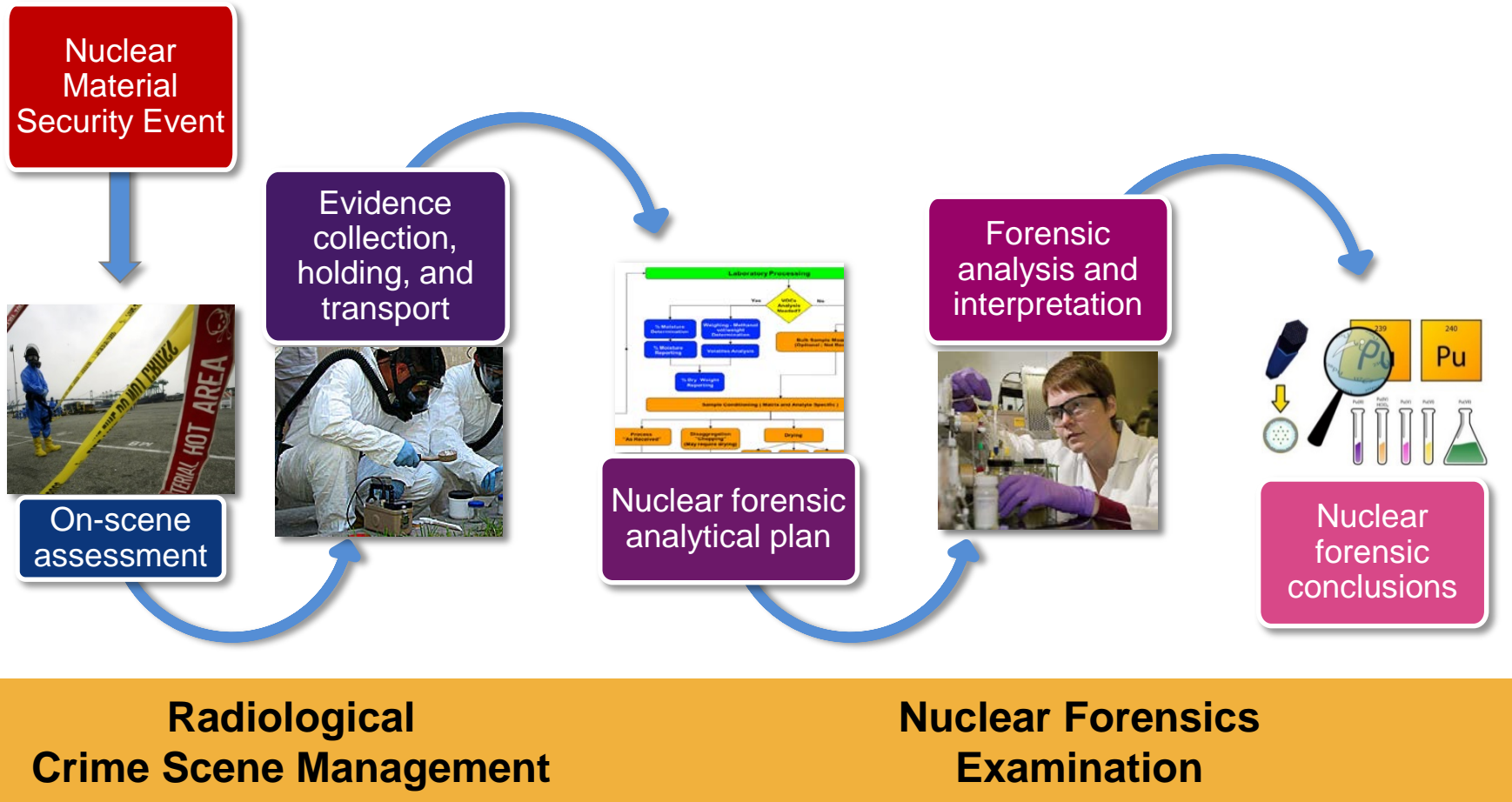
Nuclear forensic science involves the collection and examination of materials and signals, and the evaluation of data to provide investigative leads and defensible evidence regarding material and device provenance.

Nuclear security cycle



Importance of Nuclear Forensics to investigations

National response plan and nuclear forensics

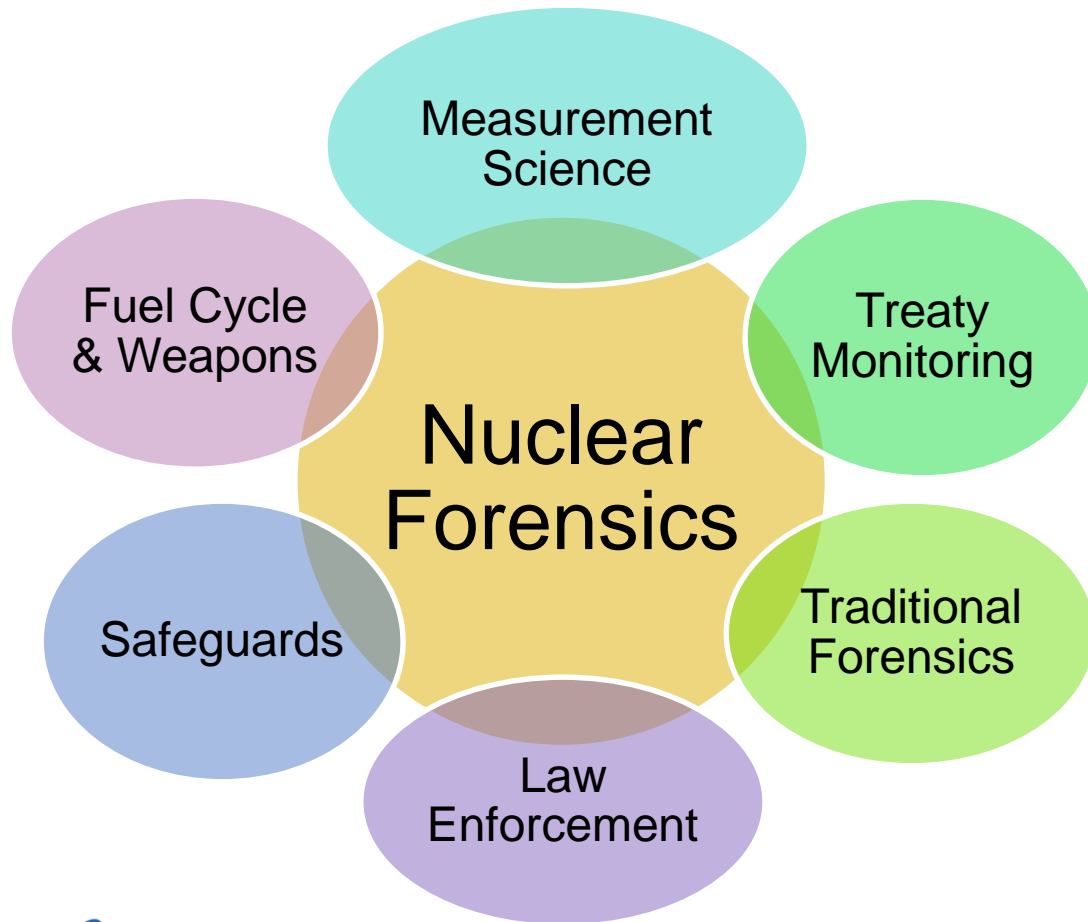


Why LANL?

- LANL expertise in radiochemistry, material science, weapons design, manufacturing, and diagnostics can trace its roots directly to the Manhattan Project
- We are the last Laboratory to retain a high degree of end-to-end capability and practiced expertise across all relevant disciplines
- World-class R&D to explore new signatures
- Capable of integrating state-of-the-art science and engineering while maintaining ties to historical baselines
- We are training a new generation across all relevant disciplines

These are the reasons the US Government looks to LANL to support the entire spectrum of nuclear forensics activities!

Nuclear Forensics Expertise

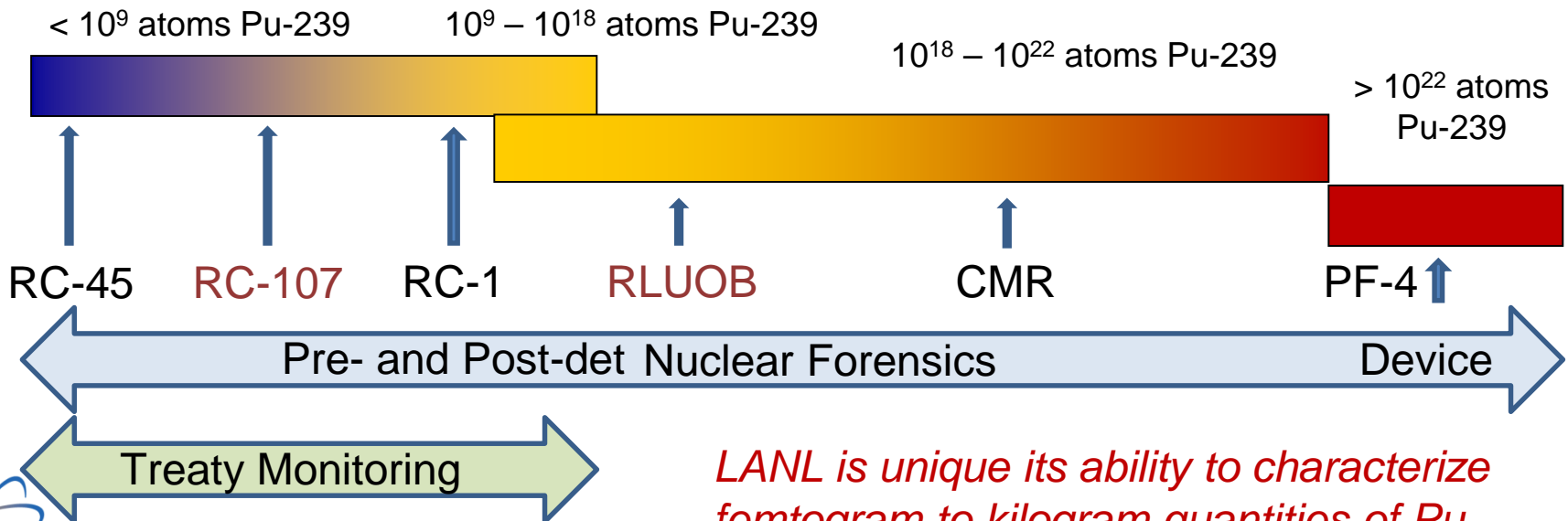


Many Disciplines Contribute

- Radiochemistry
- Geochemistry
- Analytical chemistry
- Material science
- Reactor physics
- Nuclear engineering
- Process engineering
- Enrichment engineering
- Health physics
- Statistics

Facilities supporting NF examinations

All facilities house ongoing missions that exercise analytical capabilities routinely

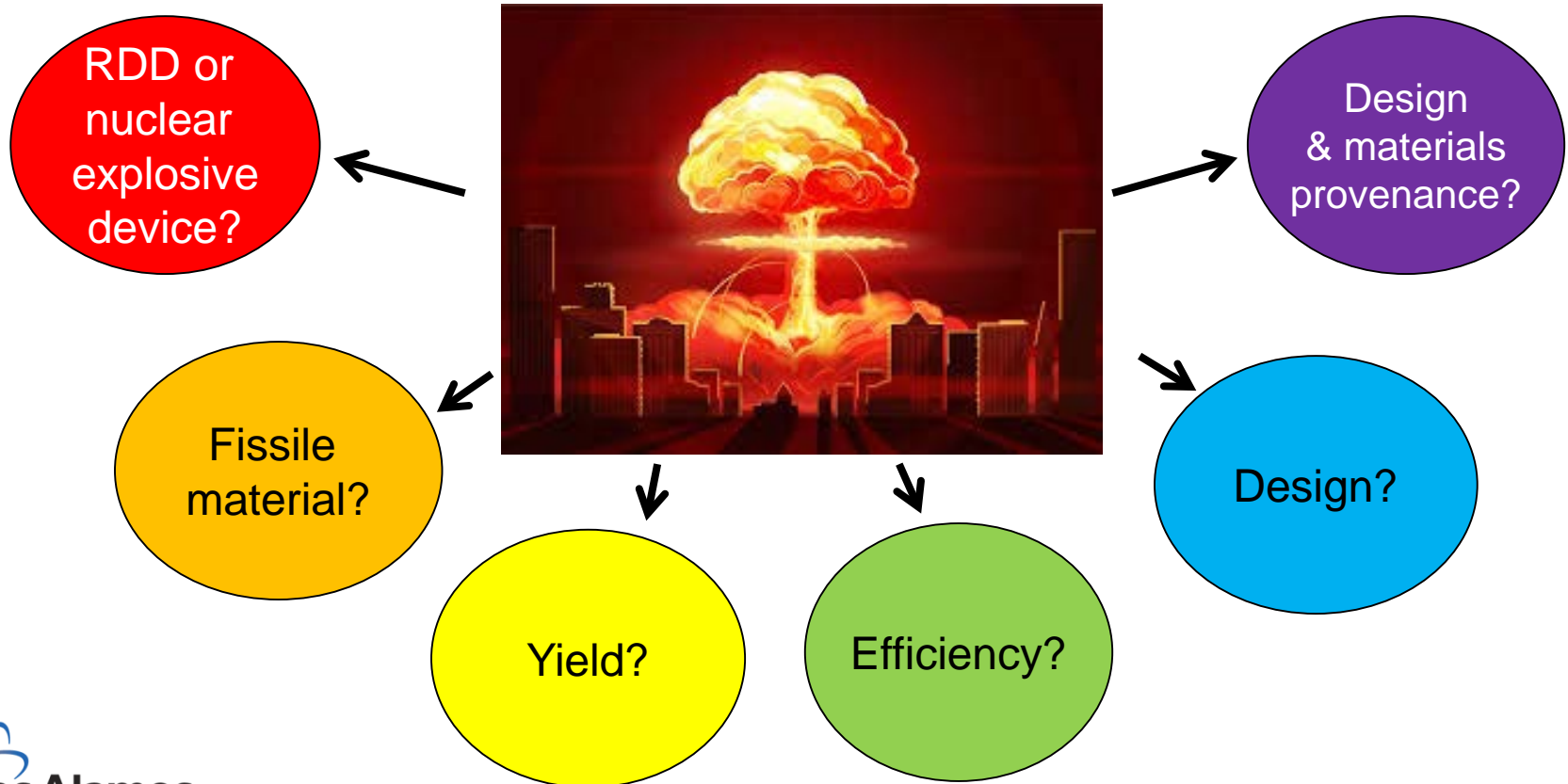


LANL is unique its ability to characterize femtogram to kilogram quantities of Pu

**Post-detonation forensics:
Investigating an act of
nuclear terrorism**

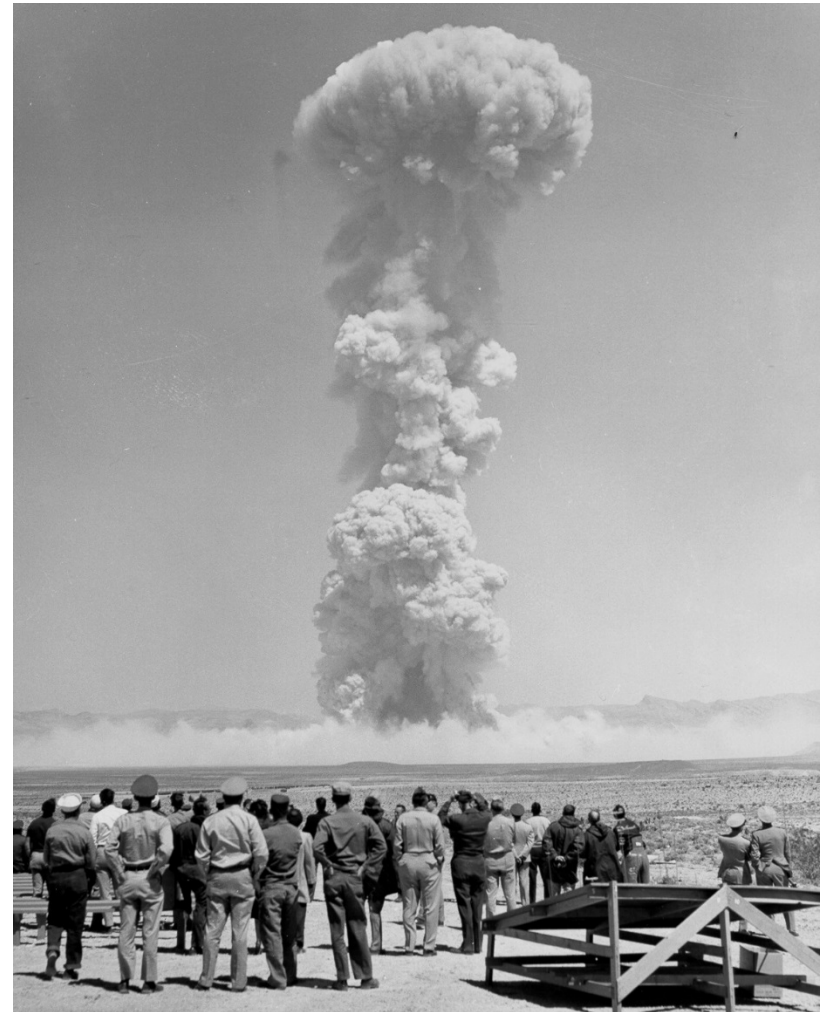
When the unthinkable happens!

- LANL is responsible for using nuclear forensics to help investigators figure out what happened



Radiochemistry ties to the US nuclear testing

- Radiochemistry used in post-detonation nuclear forensics grounded in experience from US Nuclear Test Program
 - 1) Fission products
 - 2) Short-lived actinide activation products
 - 3) Long-lived actinides



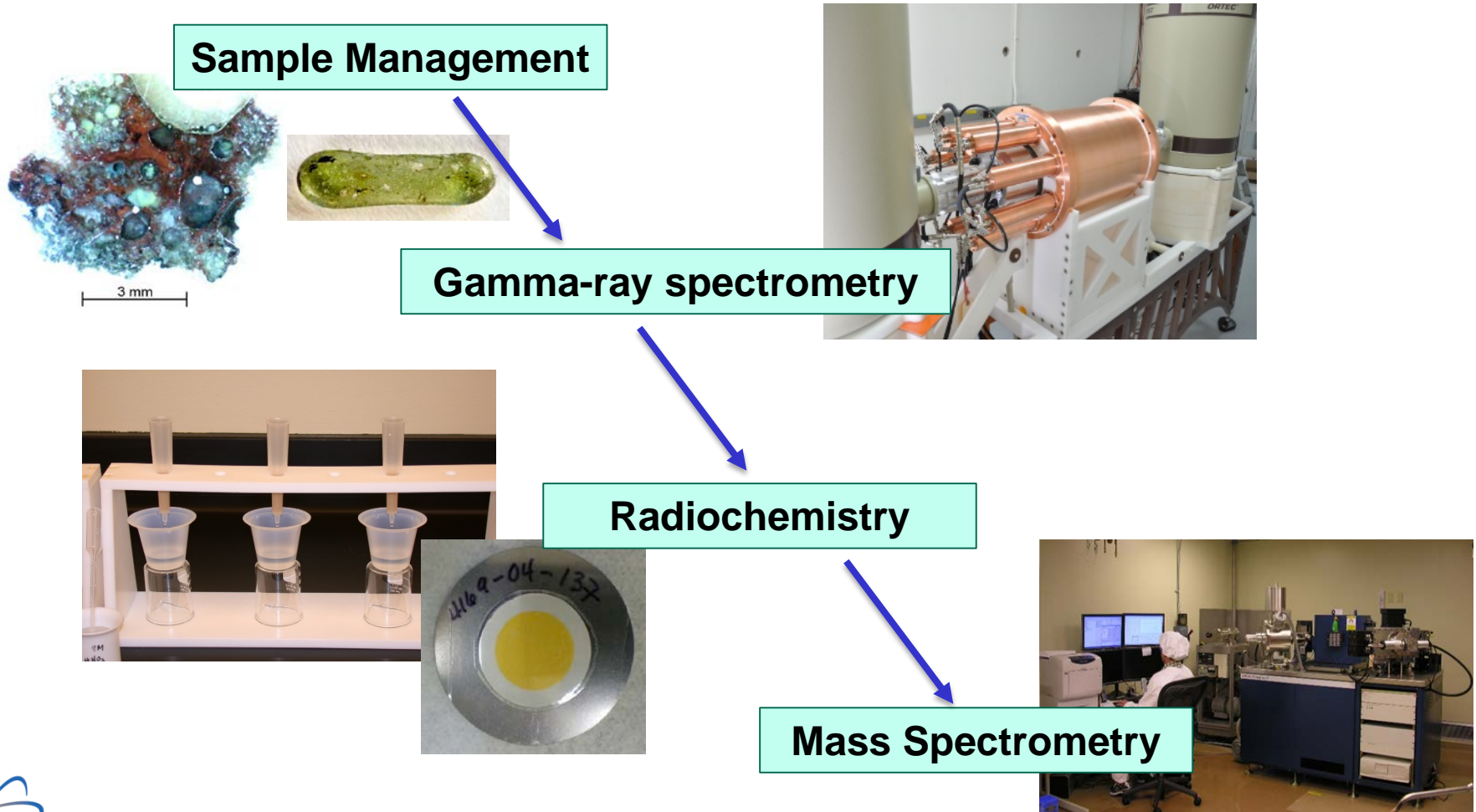
Debris Sample Collection

- Ground and air sample collections



- Army and DOE responsible for ground collection
- Air Force responsible for air collections
- Samples sent to LANL and PNNL for analysis

Characterization of debris samples



Radiochemical debris analysis

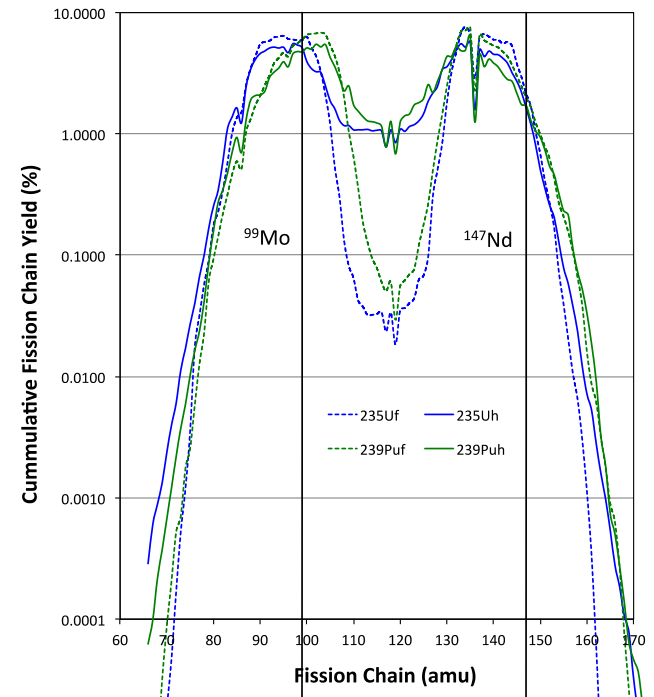
- Debris samples are completely dissolved
- Elements of interest radiochemically separated
- Radionuclides measured using gamma-ray spectrometry, beta counting, alpha spectrometry, and mass spectrometry
- Some useful isotopes include:

Fission products: ^{89}Sr , ^{95}Zr , ^{99}Mo , ^{111}Ag , ^{115}Cd ,
 ^{136}Cs , ^{140}Ba , $^{141,143,144}\text{Ce}$, ^{147}Nd , ^{153}Sm , ^{156}Eu

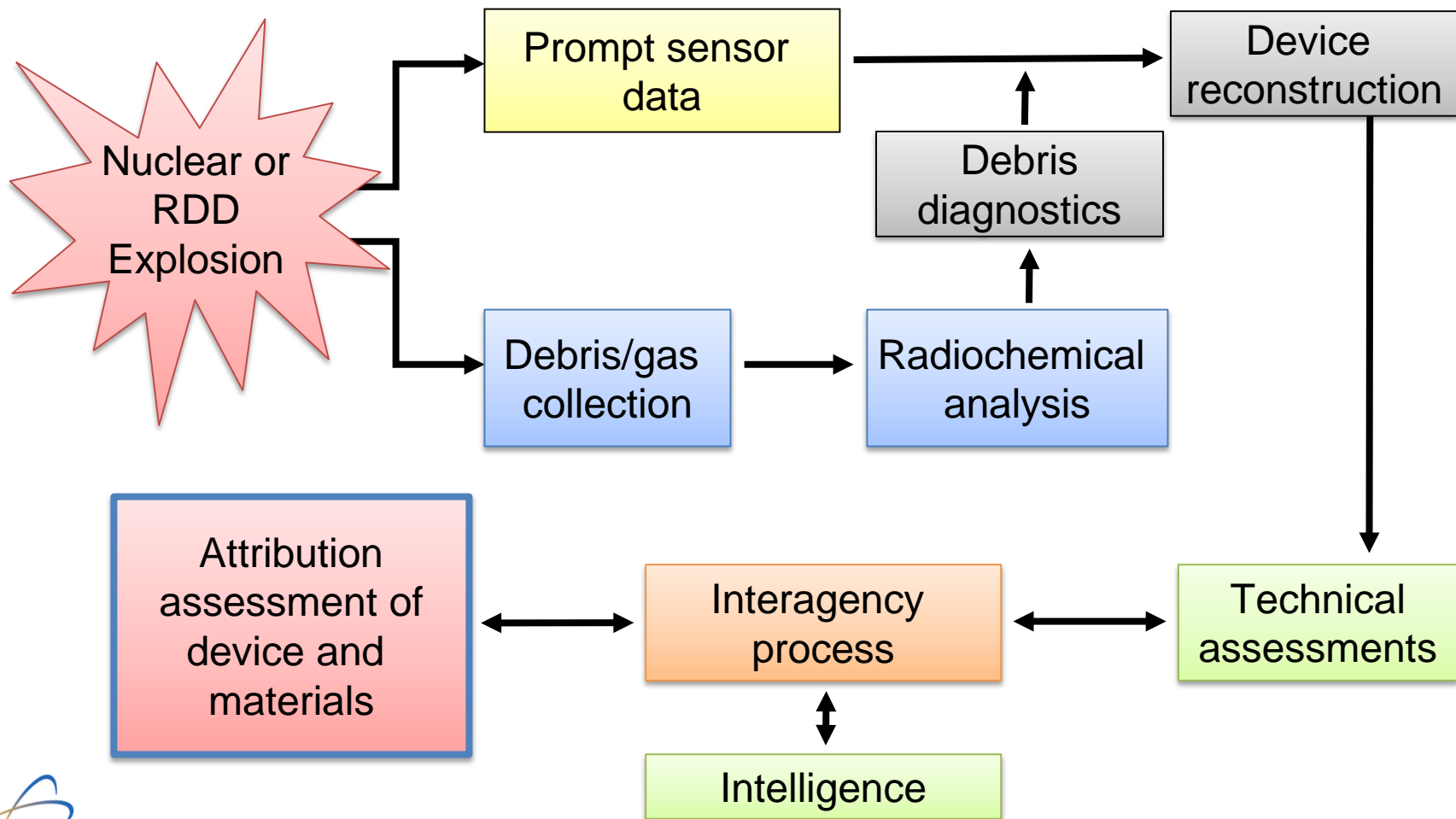
Short-lived actinides: ^{237}U , ^{239}U (^{239}Np), ^{240}Am

Long-lived actinides: ^{234}U , ^{235}U , ^{236}U , ^{238}U

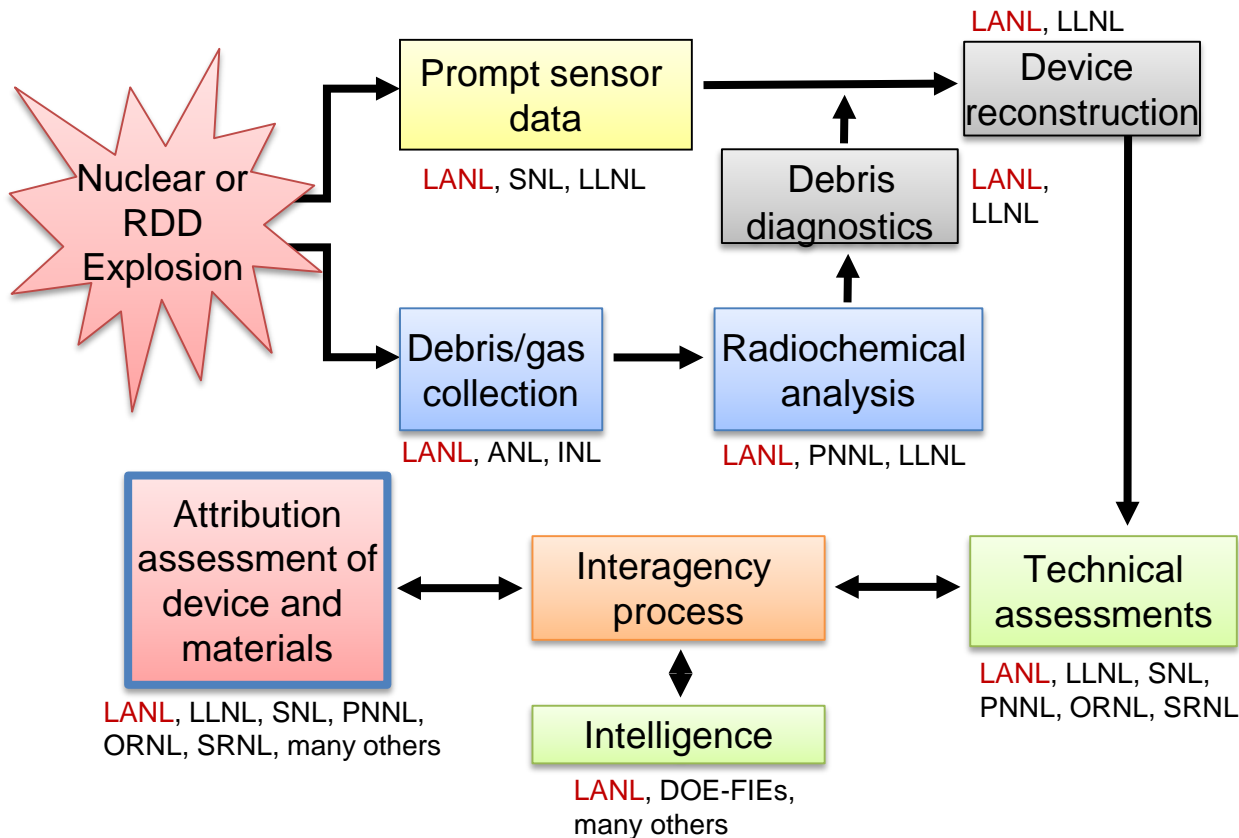
^{238}Pu , ^{239}Pu , ^{240}Pu , ^{241}Pu , ^{242}Pu , ^{241}Am



What happens next? Assessments



LANL's R&Rs in Post-det NF



LANL, unlike any other DOE laboratory, contributes to every operational aspect of post-detonation nuclear event characterization

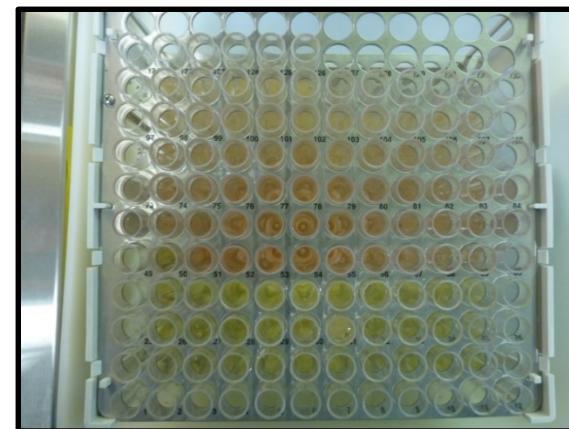
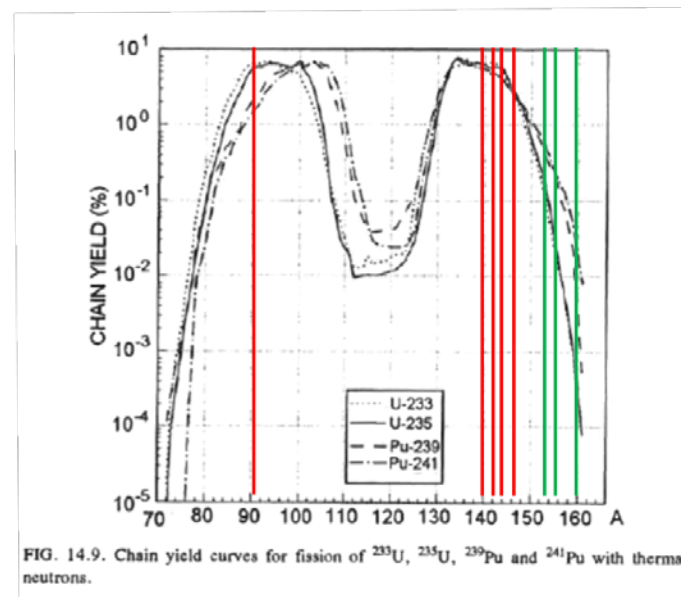
- Our operational roles are integral to our focused and relevant R&D portfolio

— NA-20s, NA-80's, DTRA, Air Force, FBI, DHS, DOE-IN, etc.

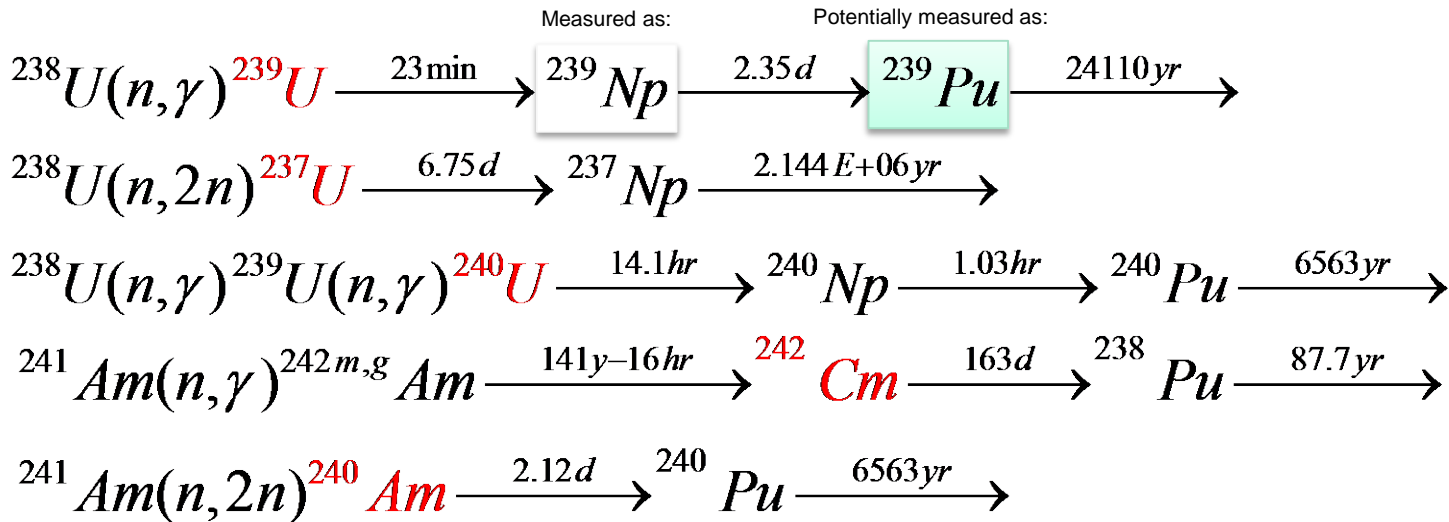
**Post-detonation forensics:
Improving measurements
through R&D**

High-efficiency HPLC based lanthanide separations

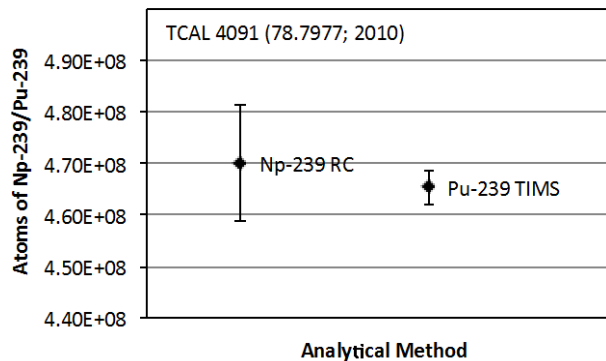
- > 40 % of fission product isotopes analyzed during a T-Cal are lanthanides
- HPLC enables rapid separation of Y, Ce, Nd, Sm, Eu, and Tb
 - High yield – ^{91}Y , $^{141/143/144}\text{Ce}$ & ^{147}Nd
 - Low yield – ^{153}Sm , ^{156}Eu & ^{161}Tb



Short-lived / long-lived progeny intercalibration

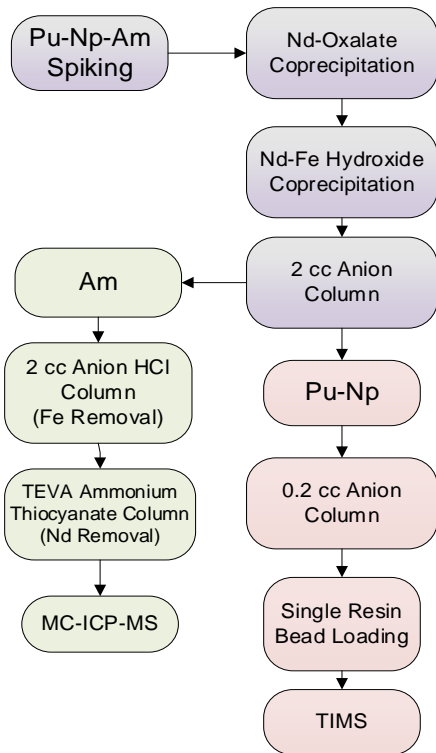


Parent / progeny intercalibration



Activity and atom scale equivalency becoming more important as we interchangeably use radiometric and mass spectrometric techniques

Sequential Pu/Np – Am radiochemistry*



Am isolated from same aliquot as Pu-Np for low-level collections

Am is measured on the Nu Plasma II multi-collector ICP-MS

Am Chemical Recoveries are 80-90%

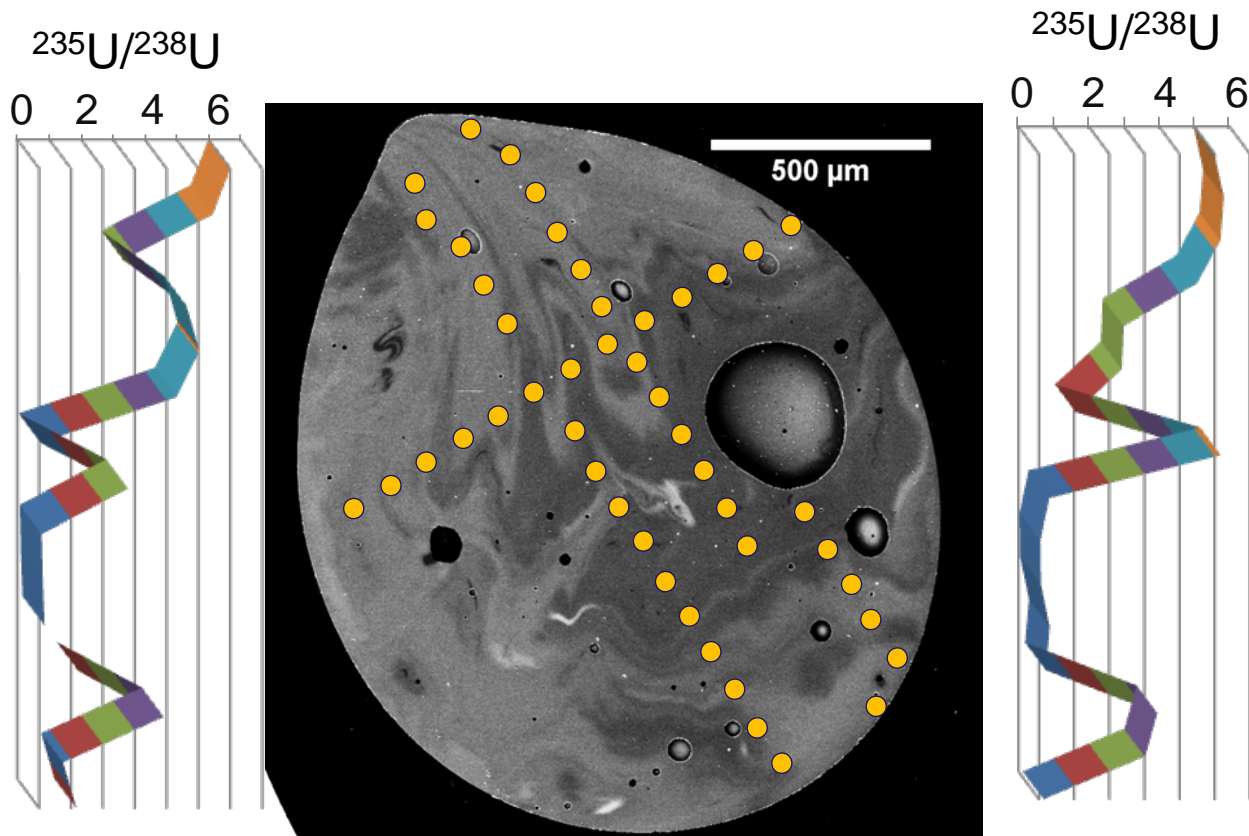
Detection limits $\sim 3E+05$ atoms Am-241/aliquot*

- Tested in this year's Long-Lived Actinide QA Round Robin

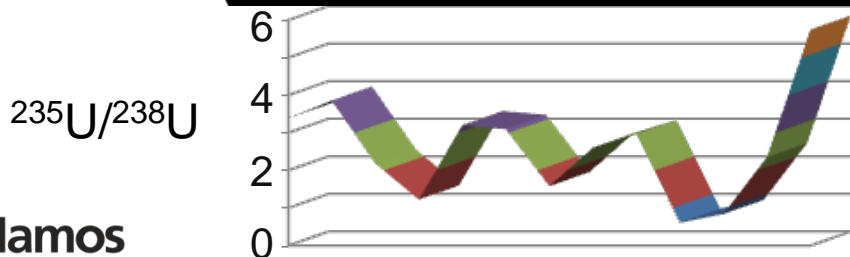
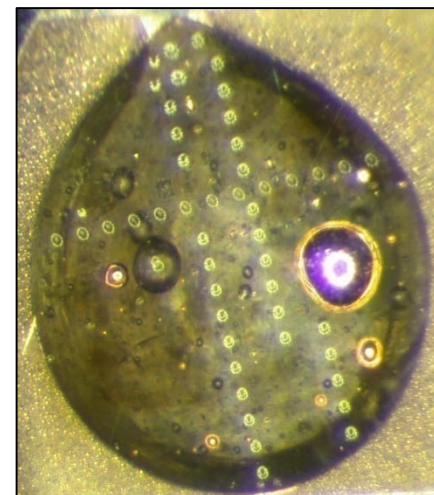


*S. Goldstein et. al., Sequential Chemical Separations and Multiple Ion Counting ICP-MS for ^{241}Pu - ^{241}Am - ^{237}Np Dating of Environmental Collections on a Single Aliquot, J. Radioanal. Nucl. Chem. 2018 (in press) LAUR-18-24606

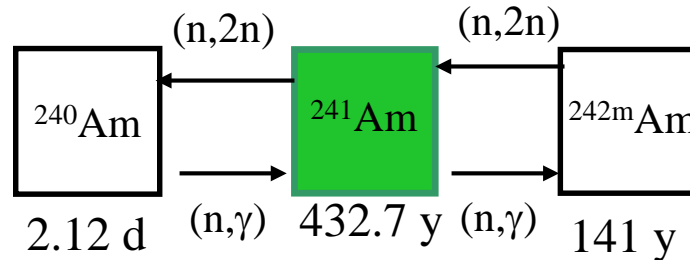
Cameca 1280 Large Geometry SIMS



- Heterogeneous!
- Brighter material has higher $^{235}\text{U}/^{238}\text{U}$ (varies by sample)



Nuclear Data R&D

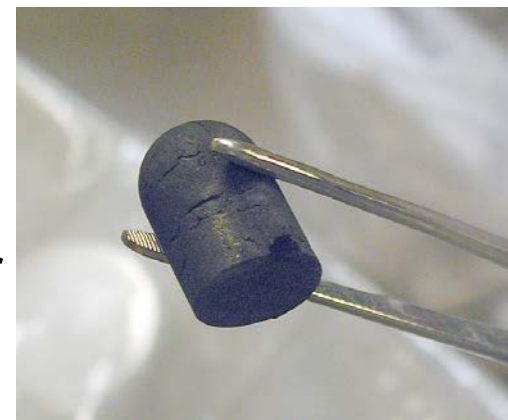


- Nuclear forensics benefits from improved nuclear data
- Reactions evaluated in recent years include: $^{234,236}\text{U}(n,g)$, $^{235}\text{U}(n,n')$, $^{235\text{m}}\text{U}$, $^{235\text{m}}\text{U}(n,\text{fission})$, $\text{Pu}(n,g)$, $^{241,242\text{m}}\text{Am}(n,g)$, and $^{241}\text{Am}(n,2n)$
- Unique combination of capabilities: radiochemistry, mass separation, low-level counting, DANCE / LSDS at LANSCE, and NCERC

**Pre-detonation forensics:
Investigating incidents
involving nuclear materials
out of regulatory control**

Pre-Detonation Nuclear Forensic Science

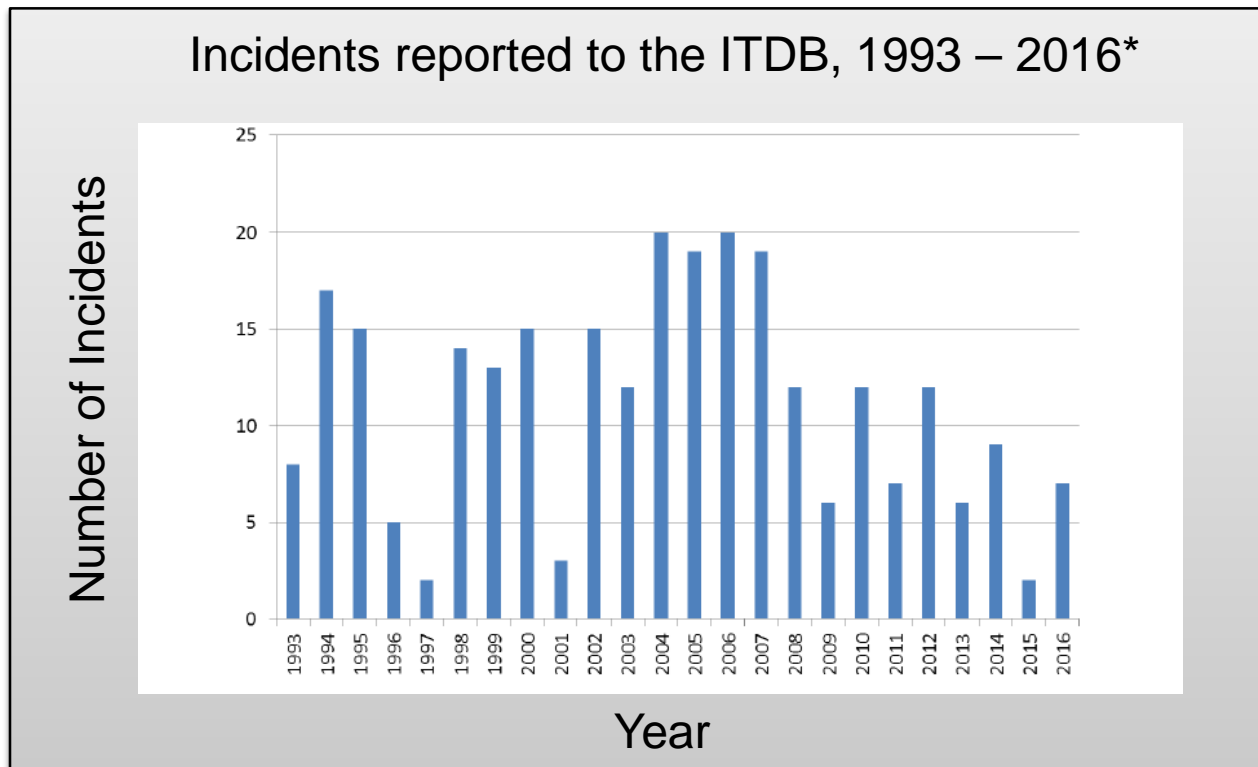
- *Nuclear forensics* is the collection and analysis of nuclear or radiological material to support investigations into the diversion, trafficking, or illicit activities involving materials
- What is the material?
- What was its intended use?
- How was the material produced?
- When was the material last processed?
- Where is the material used, produced, or stored?
- Who is associated with a material?



Goal: Link nuclear or radioactive material to people, processes, events and/or locations

Why Is Nuclear Forensics Important?

Nuclear and radioactive materials are found outside of regulatory control



* IAEA ITDB Factsheet, 2017

1999 Bulgaria 73% HEU Example

Non-nuclear forensics

Wax type

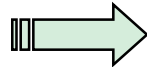
Wax colorant

Paper origin

Pb metallurgy

Pb isotopics

Ampoule material



Nuclear material forensics

Particle characterization

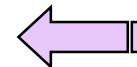
Stoichiometry

Impurity elements

Residual radionuclides

Age-dating

U & Pu isotopics

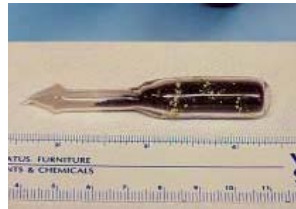


LLNL-Led Effort: Excellent demonstration of what could be done!

Nuclear Forensics Part 1: Evidence

Part 1: Traditional Forensics:
Link individuals to criminal activity

Material
Analysis



Judicial
Proceedings

Trafficker
Convicted



- Important for judicial proceedings
- Requires high-quality, legally defensible analyses
 - What is it?
 - How much is there?
- Does not require a detailed analysis of all material attributes
- *Signatures generally do not play a large role in evidence for judicial proceedings*

Nuclear Forensics Part 2: Investigations

- Detailed analysis of material attributes
- SME data interpretation
- Assessment of material process history and provenance
- Connecting material to people, places, and other materials
- *Signatures play a key role in answering investigative questions and generating investigative leads*

Part 2: Investigative Forensics: History of nuclear material

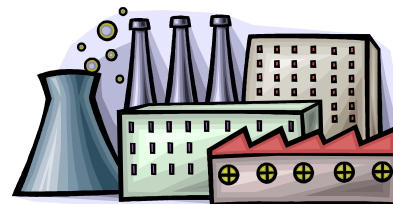
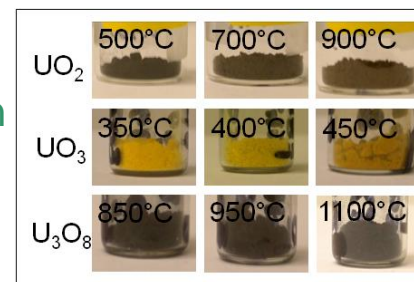


Full Characterization

- Precision isotopics
- Chemical composition
- Age dating
- Morphology

Comparative Analysis & Signature Evaluation

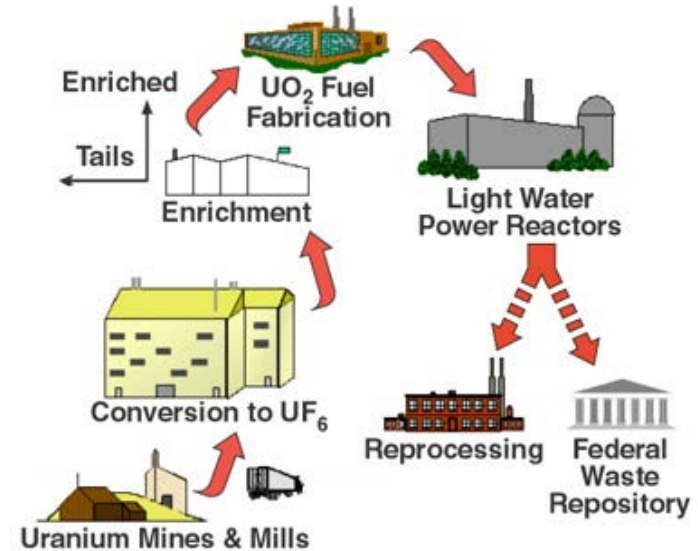
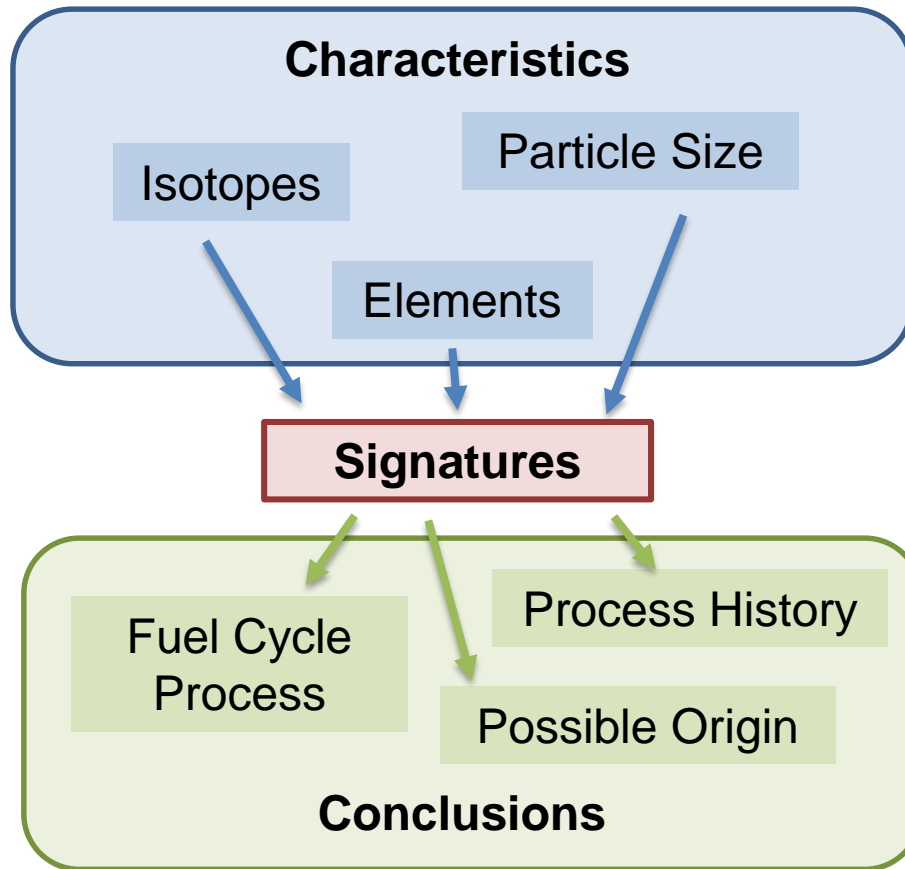
- Intended use
- Process history
- Fuel cycle information



Outcome

- Possible origins
- Connections between cases
- Enhanced security

Investigative Science of Nuclear Forensics



Investigative nuclear forensics requires a better understanding how characteristics are created, changed, and lost as materials transit the fuel cycle

Signatures are characteristics, or combinations of characteristics that help to answer investigative questions

Signatures & Forensics Questions

- Signatures are tied to the question being asked
- Value of forensic signatures changes depending on context



Q1: Is this LEU oxide powder from a LWR fuel production plant?

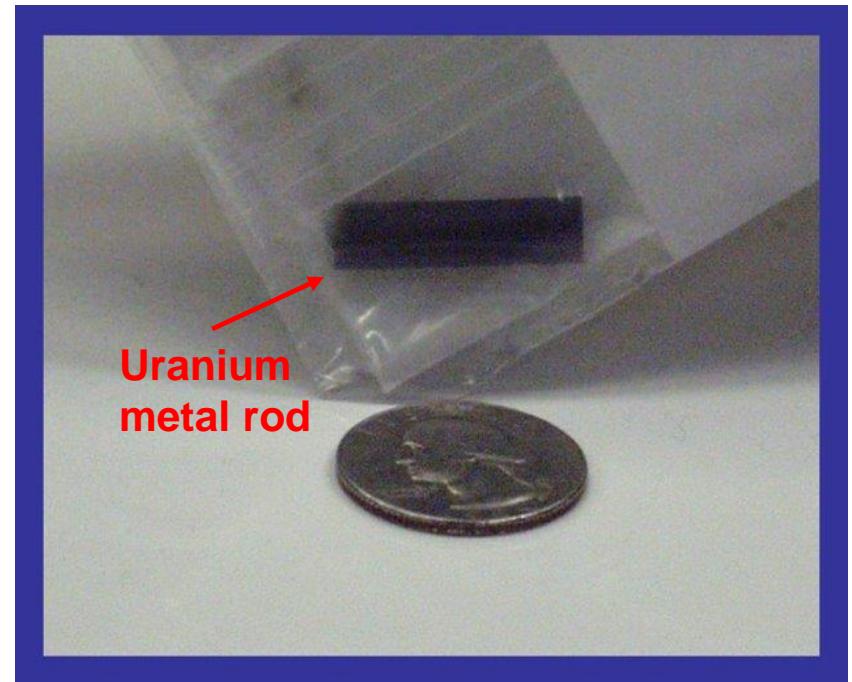
Q2: Is this LEU oxide powder from the LWR fuel plant in Country X or Y?

Characteristic	Analysis Result	Discriminating Signature?	
		Q1?	Q2?
Chemical form	UO ₂	Yes	No
Enrichment	4.3% ²³⁵ U	Yes	No
Trace elements	20 ppm Mo	No	Yes

Manhattan Project era uranium?

- Questions:
 - Is the uranium enriched?
 - Was a small uranium metal sample associated with the Manhattan Project?
- Analytical Plan:
 - Measure U and Th isotopic compositions
 - Use radiochronometry to estimate model age

Model separation dates provide forensically valuable information independent of the availability of comparative information



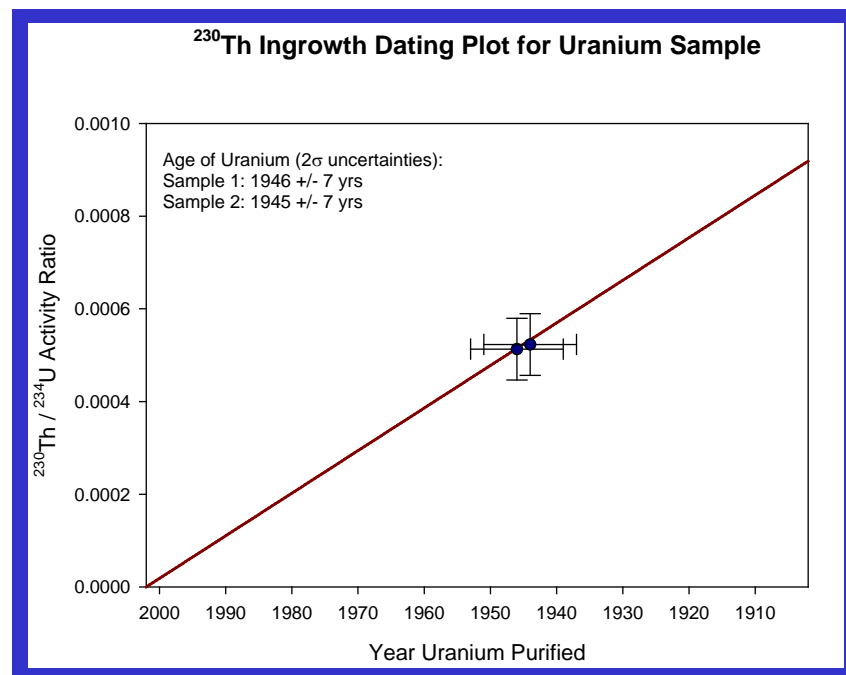
Findings from forensic examination

- U and Th concentrations and isotopic compositions determined by thermal ionization mass spectrometry

Sample Description	²³⁸ Atom %	²³⁵ Atom %	²³⁴ Atom %
Unspiked Sample	99.26 ± 0.59	0.732 ± 0.012	0.0062 ± 0.0003
Spiked Sample	99.27 ± 0.59	0.724 ± 0.012	0.0056 ± 0.0019
QC Sample (Natural U)	99.27 ± 0.35	0.724 ± 0.007	0.0056 ± 0.0005
Natural U Reference	99.275	0.720	0.0055

Conclusions provided investigators:

- The uranium is natural in isotopic composition with high confidence
- The model separation age is consistent with the Manhattan Project era with high confidence



“Is it ours?”

**The role of the national
nuclear forensics library**



Important Investigative Question: Is it ours?

- If nuclear material is found outside of administrative controls anywhere in the world, then each country should be able to answer the question:

“Is this consistent with our material?”

- IAEA guidance states that each country has a responsibility to identify materials found out of regulatory control and determine if they are consistent with those used, produced, or stored within their borders

A national nuclear forensic library (NNFL) is extremely valuable for answering this question with timeliness and confidence

National Nuclear Forensics Library Model

- A National Nuclear Forensics Library is a national system of expertise and information necessary to identify nuclear or other radioactive material found out of regulatory control

National Nuclear
Forensics Library

≠

Database

National Nuclear
Forensics Library

=

+

Forensics Experts

+

Material Experts

Data Resources

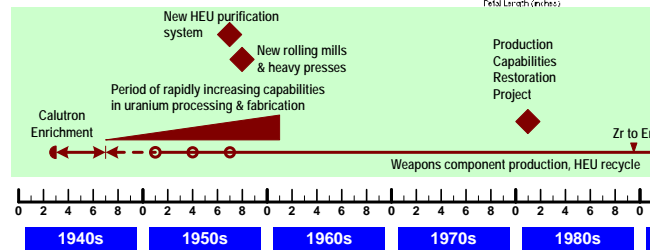
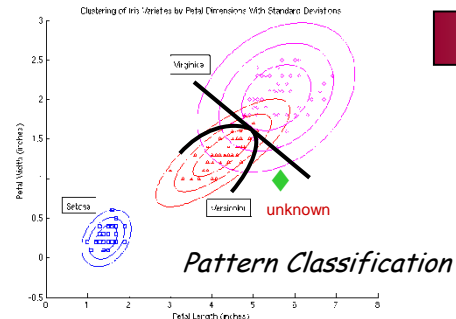
Fusion of data and expertise: NNFL

SIGNATURE ANALYSES

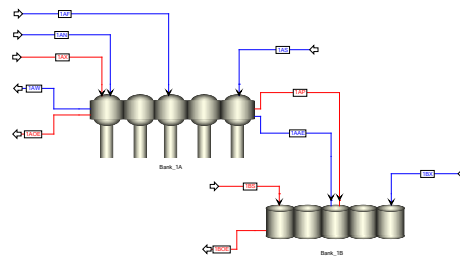
- Isotopics
- Major Elements
- Trace Elements
- Microstructure
- Morphology
- Age Dating
- Pathways Analyses



Mining & Linking Signature Data



Material Production Timelines



Signature Modeling & Validation



Capture & Linking of Expert Knowledge

EVALUATION PROCESS

TECHNICAL CONCLUSIONS

- Reveals patterns in technical nuclear forensics (TNF) data
- Resolves unanticipated and novel findings
- Enables signature discovery
- Links TNF signatures to processes, locations, facilities based on sound science

Data Evaluation and Comparative Analysis

- Three methods are commonly used to assess nuclear forensic signature data
- Point-to-point comparisons
 - Not very common, only used in special cases to link identical materials
- Point-to-population comparisons
 - Used to connect a forensic sample to a known population of materials, e.g. uranium ore concentrate from a particular mine
- Point-to-model comparisons
 - Used to identify production history and possible origins, e.g. are isotopics consistent with a particular reactor type

The need for comparative data was identified by early practitioners, who recognized the lack of complete or accessible data.

Point-to-Point Comparisons

- Used for direct comparisons, e.g.
 - Characteristics of two samples
 - Consistency of sample characteristics with technical specifications
- Statistical comparative methods

$$E_n = \frac{(A - B)}{\sqrt{(u_{c,A}^2 + u_{c,B}^2)}}$$

Where: A = reported value for Sample A
 B = reported value for Sample B
 $u_{c,A}$ = comb. std. uncertainty for Sample A value
 $u_{c,B}$ = comb. Std. uncertainty for Sample B value

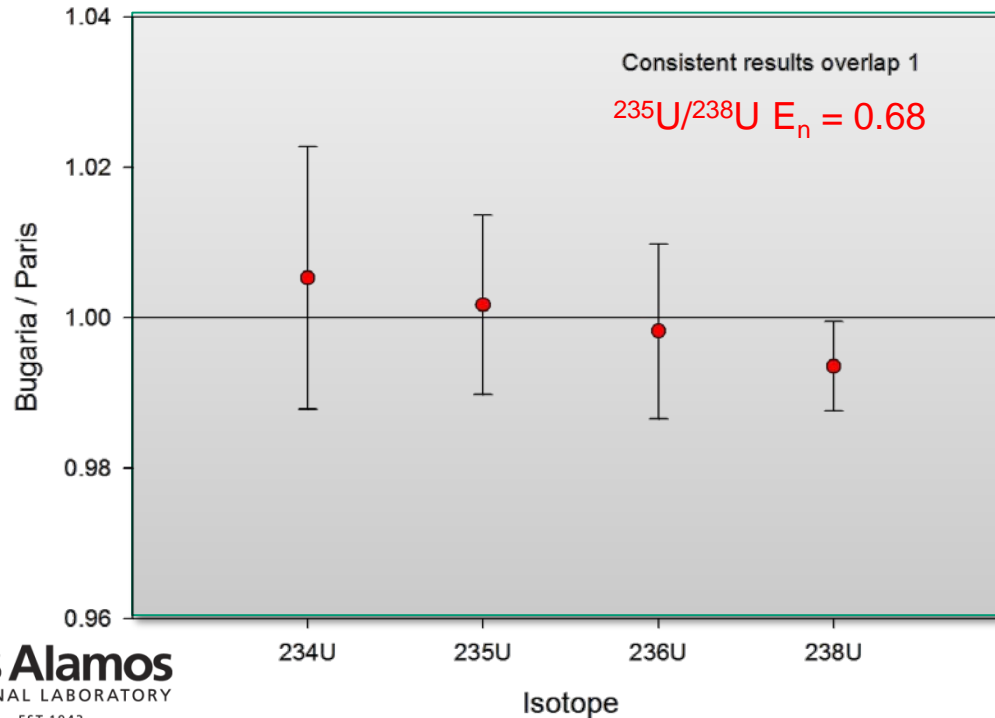
Ideal for comparisons between archived samples and unknowns

Bulgaria & France: Point-to-point example

- Small HEU sample interdicted in Bulgaria in 1999
- Similar HEU sample interdicted in France in 2001
- Were the two connected?
- Nuclear forensics used to identify similarities



Bulgaria and Paris Uranium Isotopic Composition Results



Bulgaria & France: Point-to-point example

- Thorough investigative technical nuclear forensic analysis performed on both materials
 - Bulgarian sample analyzed by DOE, French sample by CEA
 - Complete analysis of uranium
 - Associated packaging also analyzed

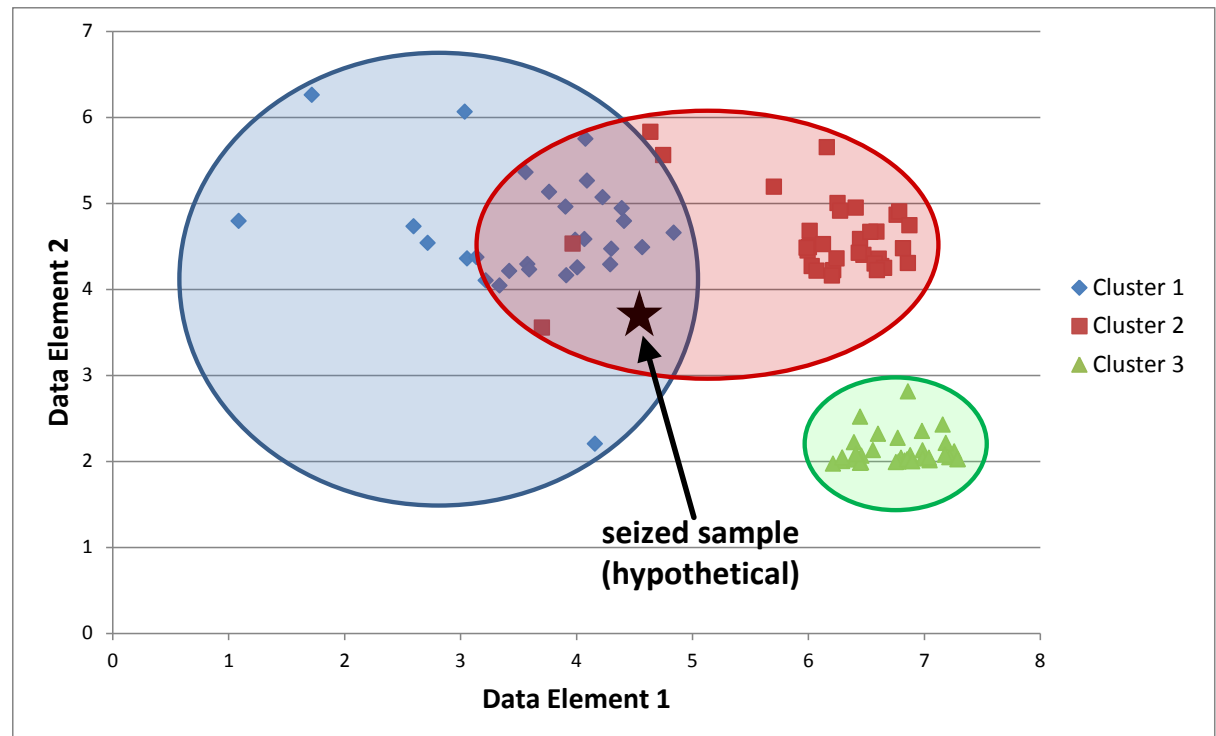
Signature	Similar?	Confidence?
Uranium Isotopic Composition	YES	High
Trace Element Concentrations	NO	Low
Material Model Age	YES	Medium
Estimated irradiation history	YES	High

Conclusion: The materials seized in Bulgaria and Paris were linked, with high confidence.

Point-to-population: when there isn't an exact match

- Most common situation, i.e. no exact match available
- Used to tie forensic samples to historic data (e.g. production measurements)
- Statistical methods can evaluate similarities using one or many characteristics

Three Material Populations



*Limitation:
conclusions are
limited by availability
of comparative data*

Slovakia UOC – Point-to-population

- Uranium sample seized in Slovakia in November 2007
- 2012 US asked to assist with forensic examination of material
- Question: Could we help identify the provenance of the UOC?

UOC Material Seized in Slovakia in November 2007



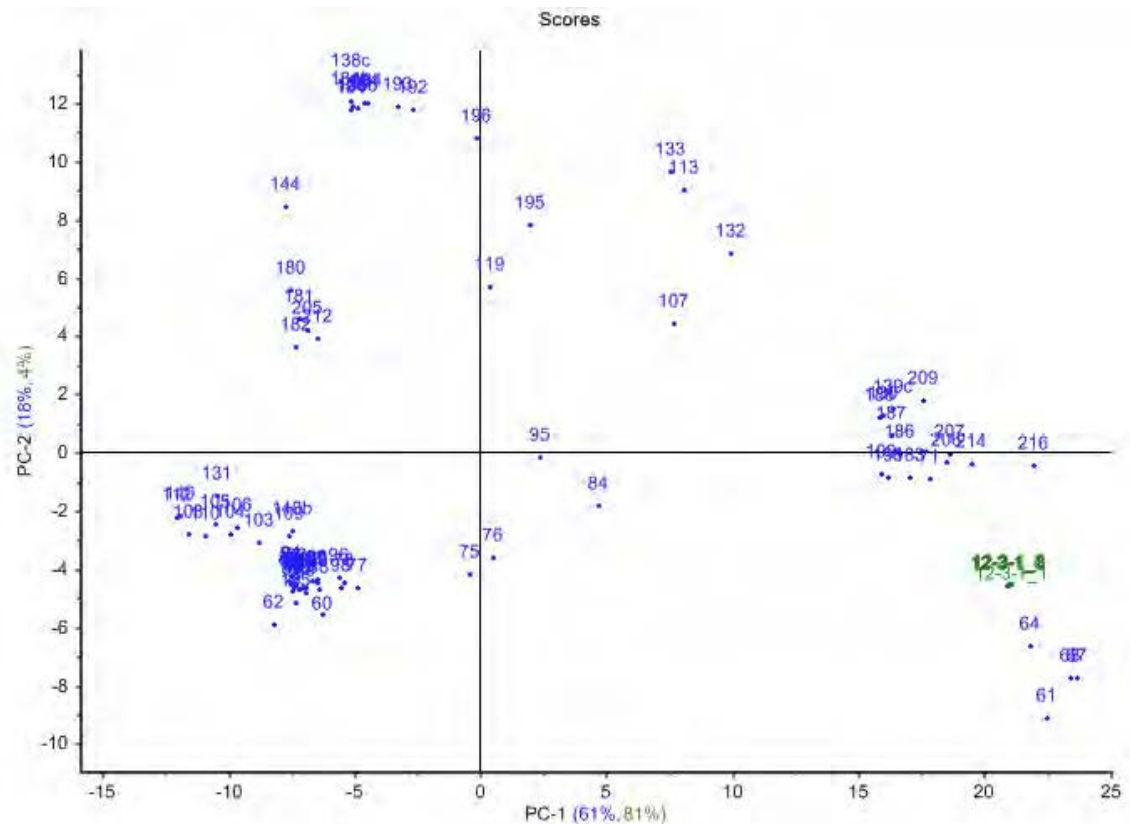
Slovakia UOC – Point-to-population

- Trace elements used to compare Slovakia sample to known populations



- The Slovakian UOC sample was not consistent with population of material represented in the UOC database

Projection of sample 12-3-1 on NIR1 score plot

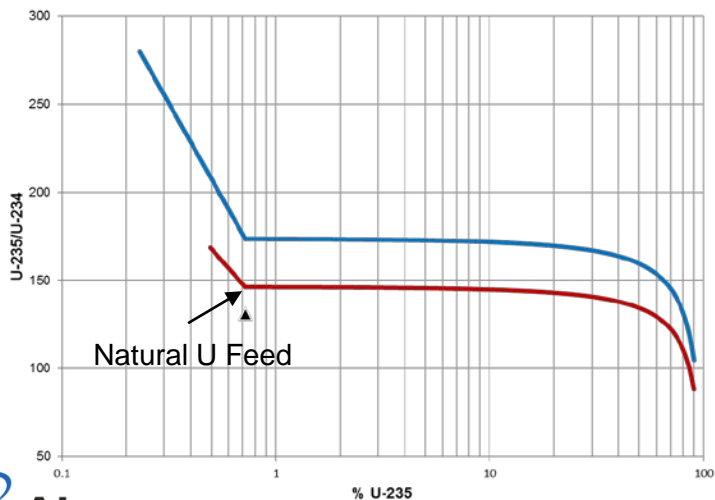


Point-to-model: using predictive models

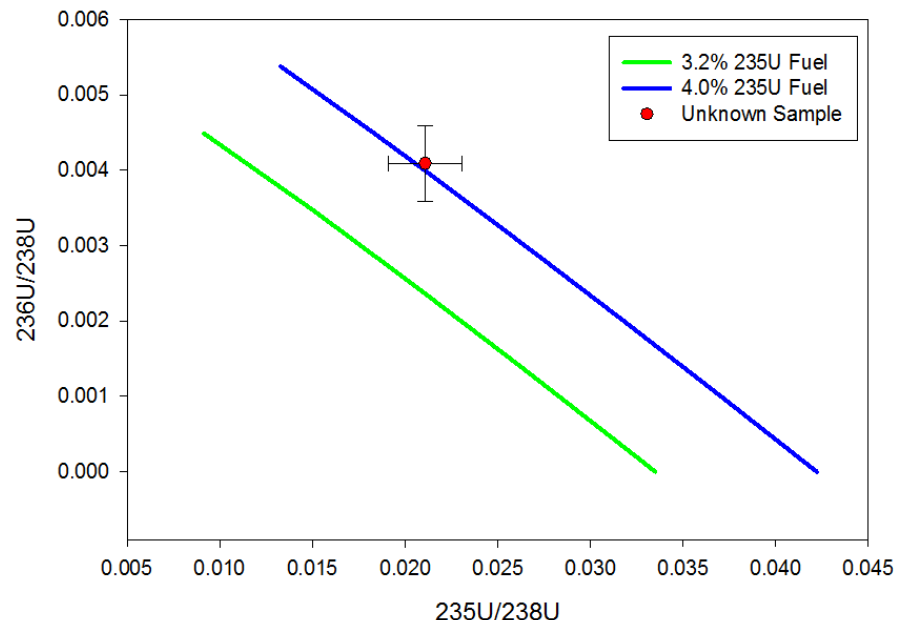
- Predictive models useful when no comparative data exists
- Can also be used to fill gaps in empirical data
- Reactor and enrichment modeling are most often used predictive models

Limitation: Predictive models generally require empirical data for validation (e.g. SFCompo)

Ideal Cascade for HEU Production $^{234}\text{U}/^{235}\text{U}$ Ratio



Comparison of Unknown Uranium Sample to Modeled PWR Burnup Data



Irradiated Material History – Point-to-model example

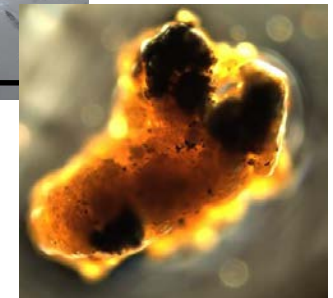
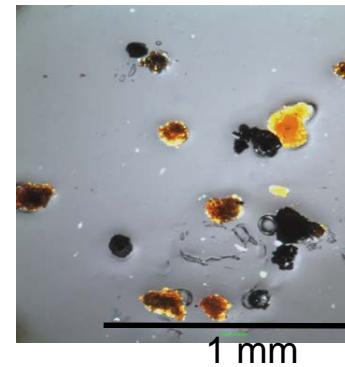
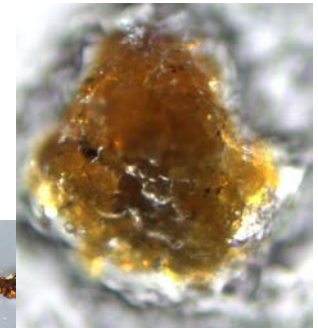
- Question: Were irradiated uranium samples recovered the same samples referenced in irradiation records?
- Gamma-ray spectrometry revealed both ^{152}Eu and ^{154}Eu activity – potentially useful signature

^{150}Eu 36.9 Y ϵ : 100.00%	^{151}Eu $\geq 1.7\text{E}+18$ Y 47.81% α	^{152}Eu 13.528 Y ϵ : 72.10% β^- : 27.90%	^{153}Eu STABLE 52.19%	^{154}Eu 8.601 Y β^- : 99.98% ϵ : 0.02%	^{155}Eu 4.753 Y β^- : 100.00%
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- Approach: Use a combination of radiochronometry and reactor modeling to assess consistency with investigative information

Calculating Reactor Irradiation Dates

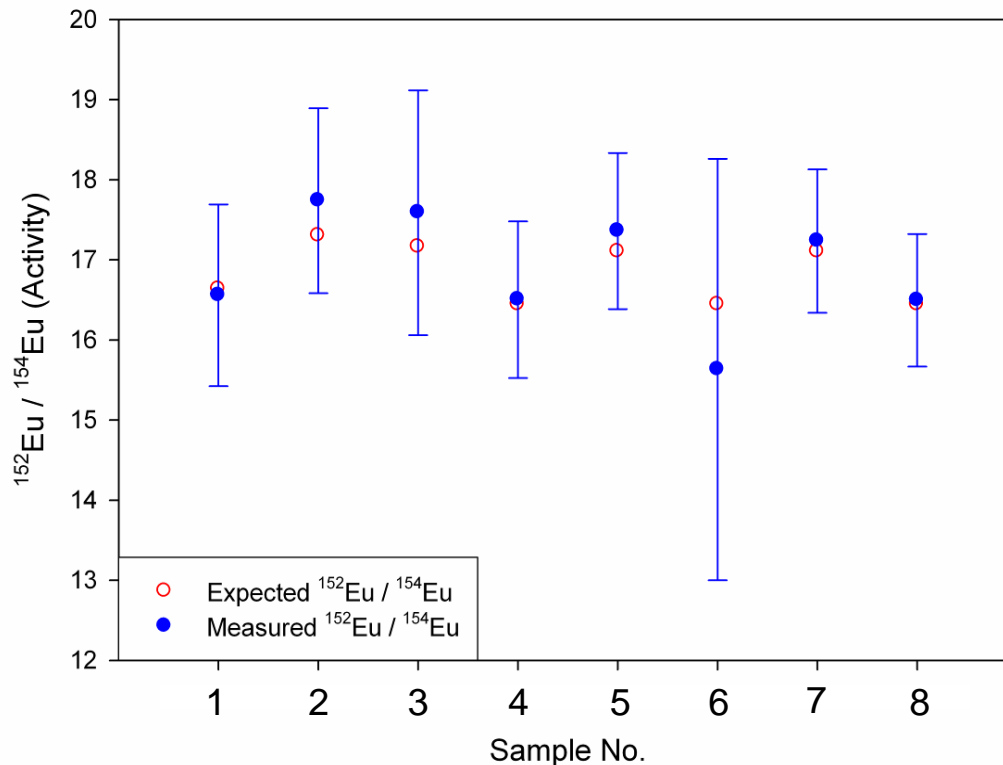
- Methodology for Calculating Irradiation Dates
 - model $^{152}\text{Eu}/^{154}\text{Eu}$ ratio
 - decay corrected ratio to analysis date
 - compare decay corrected ratio to calculated ratio
- Europium Activation
 - $^{151}\text{Eu} = 47.81\%$ $^{153}\text{Eu} = 52.19\%$
- Assumptions
 - Natural Isotope Abundance
 - Activity is from activation, not fission
 - ^{152}Eu activity 10^6 higher than expected from fission
 - ^{154}Eu activity 10^4 higher than expected from fission



Comparing measured and modeled data

Conclusion: $^{152}\text{Eu} / ^{154}\text{Eu}$ ratios consistent with declared irradiation dates with high confidence

Comparison of Measured $^{152}\text{Eu} / ^{154}\text{Eu}$ to Expected
(Irradiation Position 1: $\phi_e / \phi_t = 0.253$, Irradiation Position 2: $\phi_e / \phi_t = 0.227$)



**Recent nuclear forensics
projects: Exploring new
signatures and
identifying R&D needs**

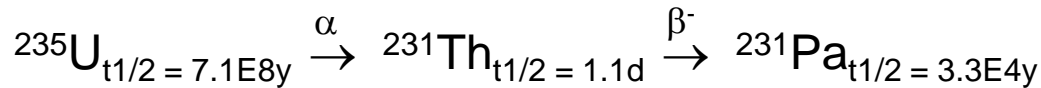
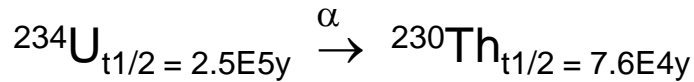
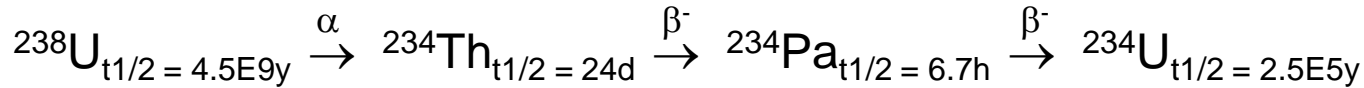
Exploring new signatures & the U.S. NNFL

- The U.S., through its national nuclear forensics library sponsors the forensic examination of materials to:
 - Build a comparative data set necessary to identify U.S. produced materials
 - Facilitate a better understanding of discriminating signatures
- Relies on characterizing materials with known process histories

NOTE: The characteristics of these materials desired for their intended use may be less discriminating than characteristics; i.e. signatures require going beyond technical specifications

Radiochronometry: A unique intrinsic signature

- Uranium decay:



Progeny / Parent Pair	Useful Time Range
$^{234}\text{Th} / ^{238}\text{U}$	0 – 7 months
$^{230}\text{Th} / ^{234}\text{U}$	> 2 months
$^{231}\text{Pa} / ^{235}\text{U}$	> 4 months

Model separation dates provide forensically valuable information independent of the availability of comparative information

Important Assumptions

- Radiochronometry provides a “model age”
 - Assumes complete parent / progeny separation at t_0
 - Assumes a closed system
- Multiple chronometers may not give the same model age
 - Discordant chronometers can provide insights into process history

Simplified ^{234}U - ^{230}Th Age Equation

$$t_{(\text{years})} = \frac{1}{\lambda_{234\text{U}} - \lambda_{230\text{Th}}} * \ln \left[\frac{R(\lambda_{234\text{U}} - \lambda_{230\text{Th}})}{\lambda_{234\text{U}}} \right]$$

$R = ^{230}\text{Th}/^{234}\text{U}$ atom ratio

$$\lambda_{234\text{U}} = 2.83 \times 10^{-6}$$

($T_{1/2} = 245,250$ (490) years)

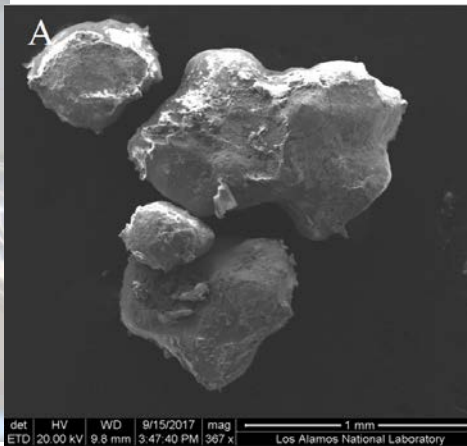
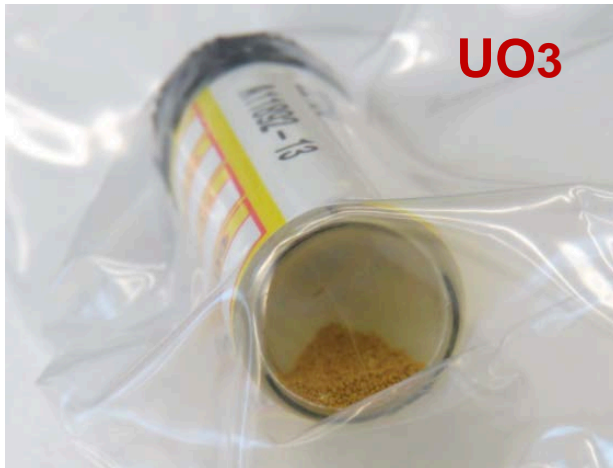
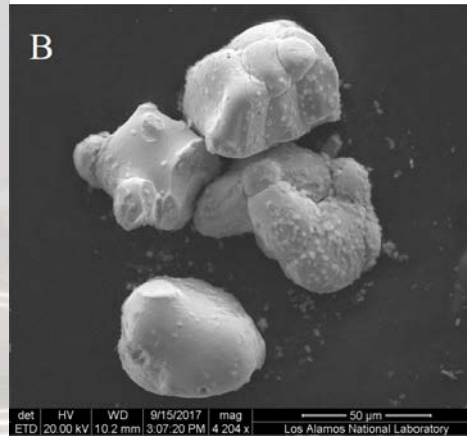
$$\lambda_{230\text{Th}} = 9.16 \times 10^{-6}$$

($T_{1/2} = 75,690$ (230) years)

Cheng, H., Edwards, R.L., Hoff, J., Gallup, C.D., Richards, D.A., Asmerom, Y. (2000)
The half-lives of uranium-234 and thorium-230. *Chemical Geology*, 169, 17-33.

The uncertainties expressed for the half lives of U-234 and Th-230 are expressed as 2σ expanded uncertainties. For error propagation purposes, the 1σ uncertainty should be used.

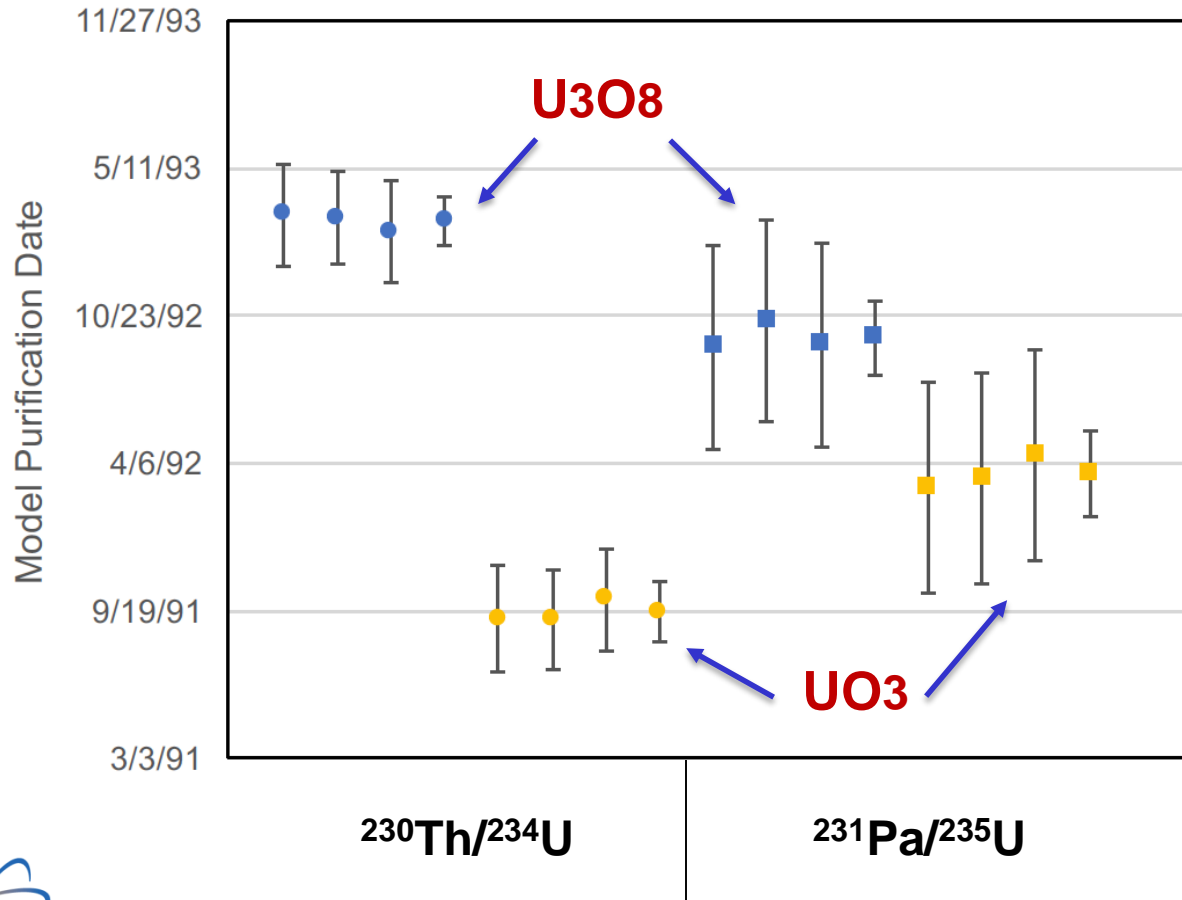
Case 1: Two US-produced uranium oxides



- Both precursors for nuclear fuel production
- Both produced at approximately the same time
- Clear differences in color, particle size, and morphology

Case 1: Radiochronometry

Model Separation Dates for Two Radiochronometers



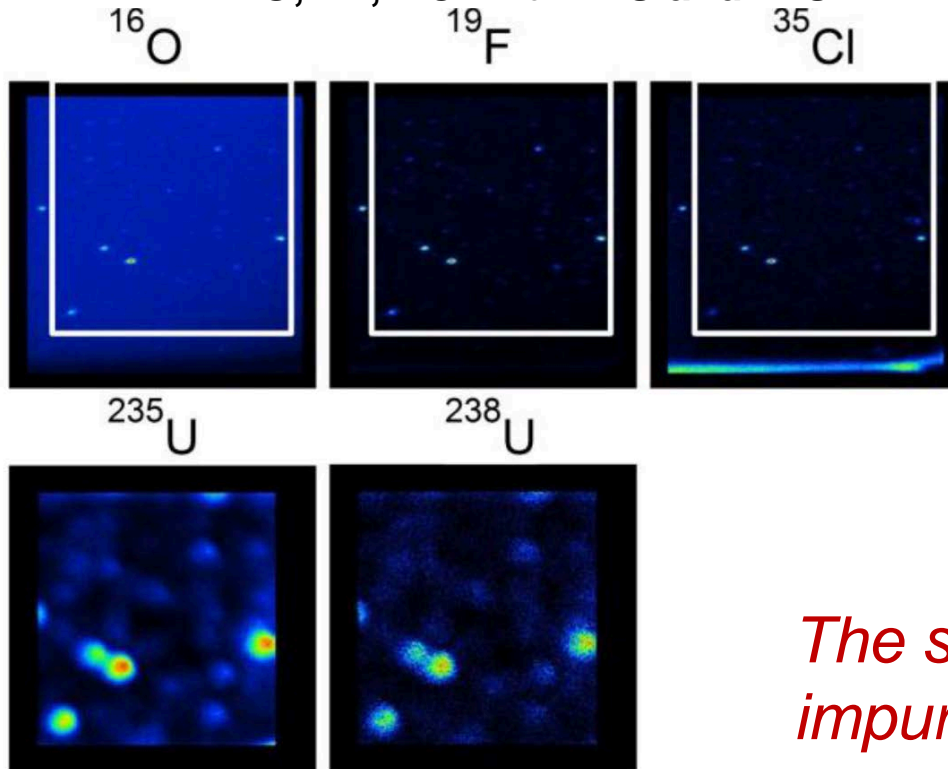
Two chronometers concordant within each material

Two materials differ in $^{230}\text{Th}/^{234}\text{U}$ age

Case 1: Spatially resolved signatures

SIMS: Halogens and Light Isotopes

^{16}O , ^{19}F , ^{35}Cl with ^{235}U and ^{238}U

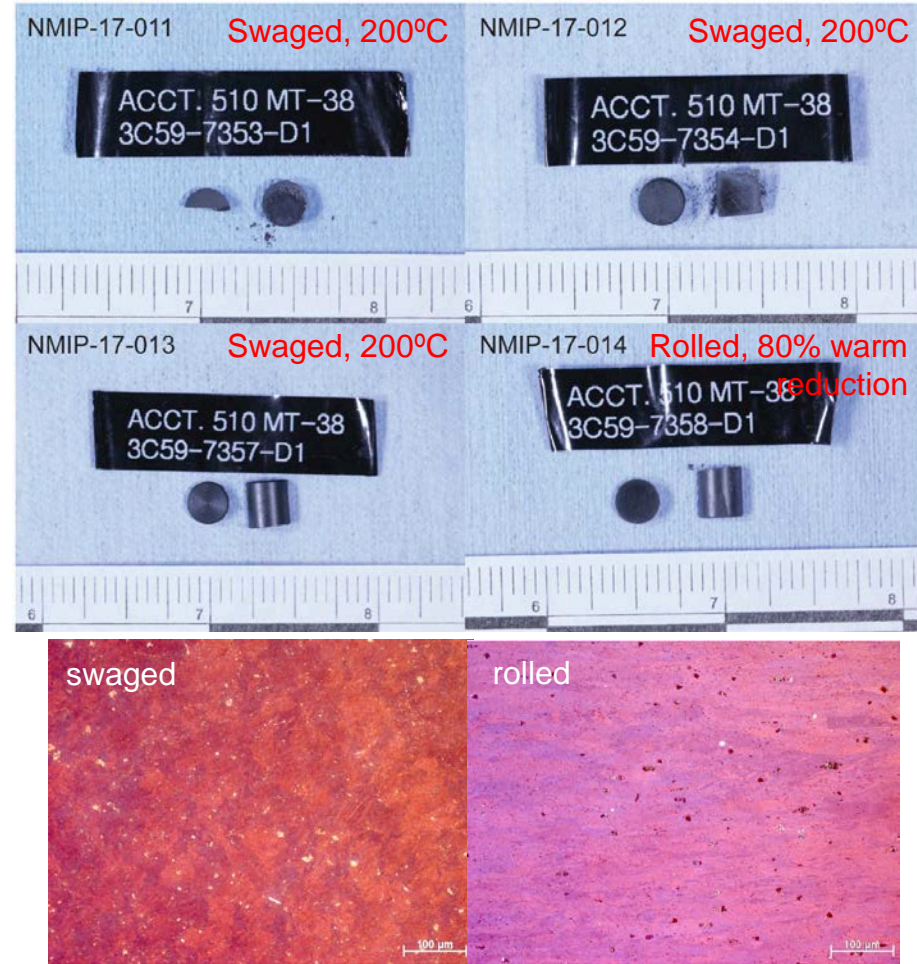


- UO_3 sample has F and Cl associated with U in particles
- U_3O_8 sample contained F and Cl, but only as discrete salt particles

The spatial distribution of impurities may be a more important signature than concentration

Case 2: Four U.S. uranium metal samples

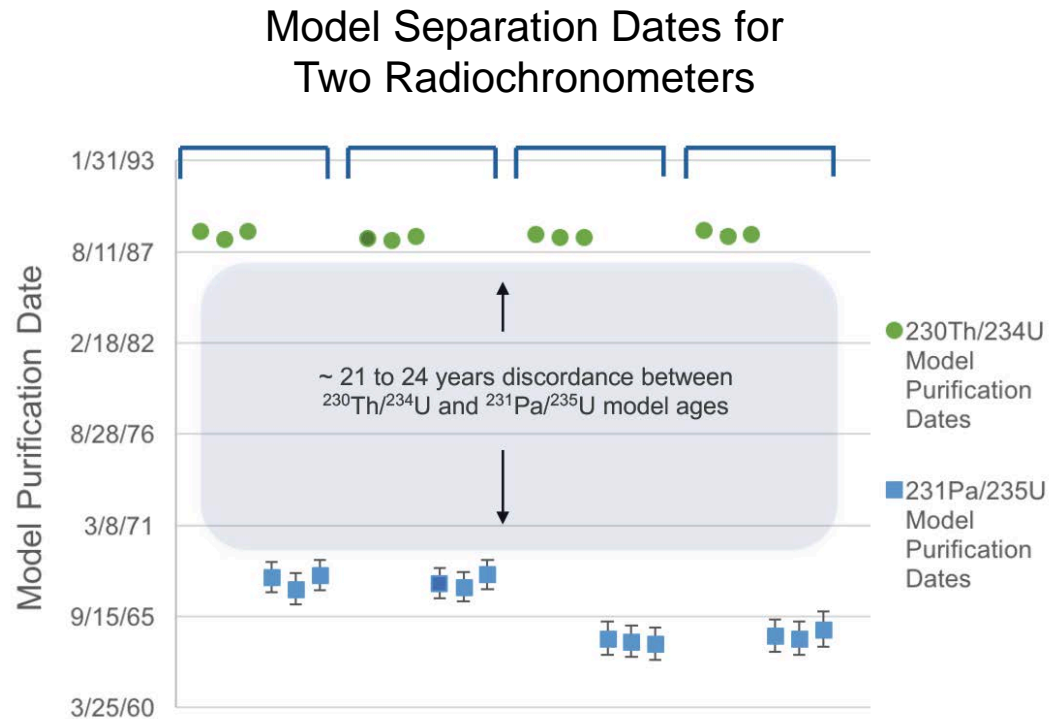
- Metal representative of late 1980's U.S. U metal
- Chemically and isotopically similar, but processed differently
 - 3 swaged cylinders
 - 1 rolled cylinder
- Different grain geometries ties material to process method



Rolled metal shows directionally elongated grains

Case 2: Radiochronometry

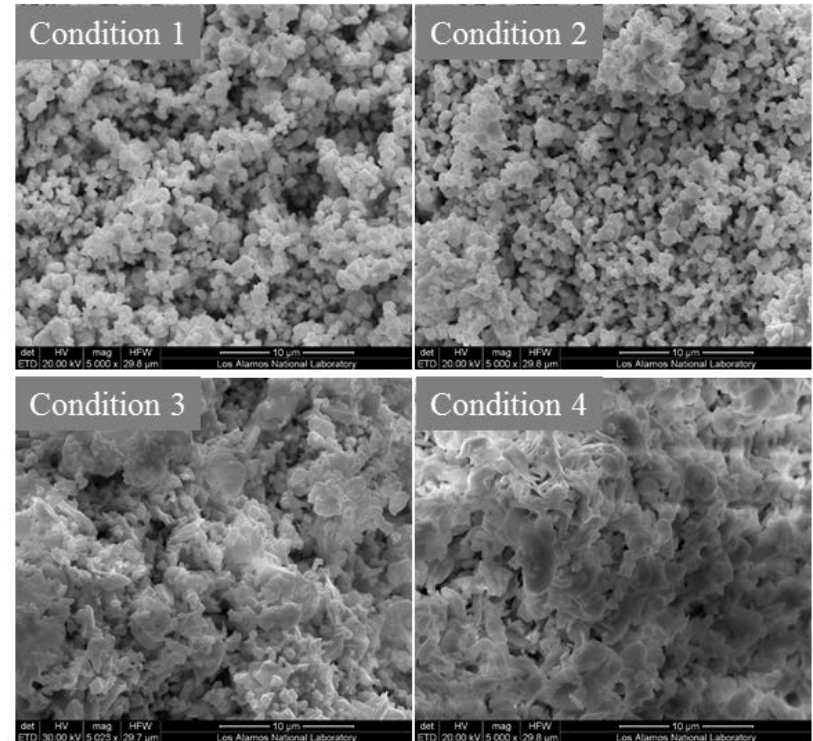
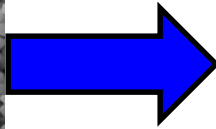
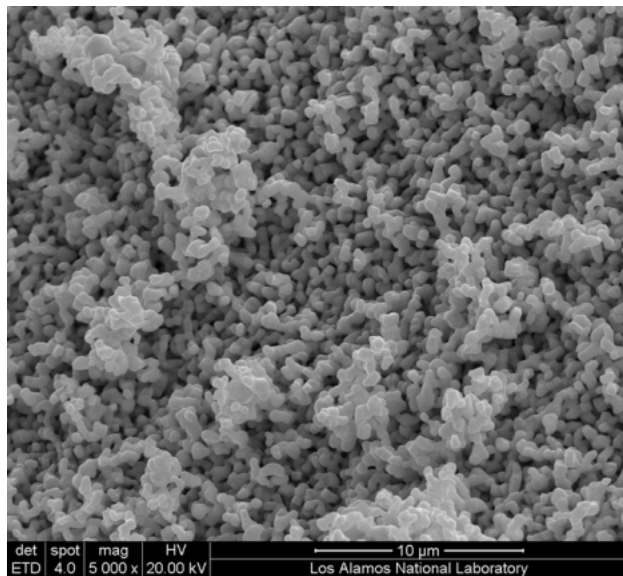
- Two chronometers discordant
- Metal cast sometime in the late 1980's
- Uranium chemically processed sometime prior, but not before the mid-1960's
 - Based on observations for other metal and oxide samples



Hypothesis: Thorium is more efficiently purified from uranium than protactinium during casting operations.

Case 3: Temporal morphology changes in $\alpha\text{-U}_3\text{O}_8$

SEM images of $\alpha\text{-U}_3\text{O}_8$ following storage under controlled conditions for two years



Changes in morphology were observed following storage under high humidity conditions (~ 90% relative humidity)

Nuclear forensics: International outreach

Nuclear security and MORC are global issues

- Illicit trafficking of nuclear materials is, by nature, transnational problem
- IAEA guidance recommends all countries have a national response plan for nuclear security events that includes responding to incidents involving MORC
- Many other countries have, or are developing nuclear forensic capabilities
- The U.S. strategically engages with other countries
 - Capacity building for nascent programs
 - Peer-to-peer work to further the science of NF



Capacity building

- Bilateral and multilateral engagement to build awareness and basic nuclear forensics capabilities
 - Establish best practices
 - Connect technical and law enforcement communities
 - Laboratory training in basic nuclear forensic examination techniques



Gamma-ray spectrometry training at LANL for South Africa, Estonia, and Georgia



Alpha spectrometry training on-site in Armenia



Peer engagement

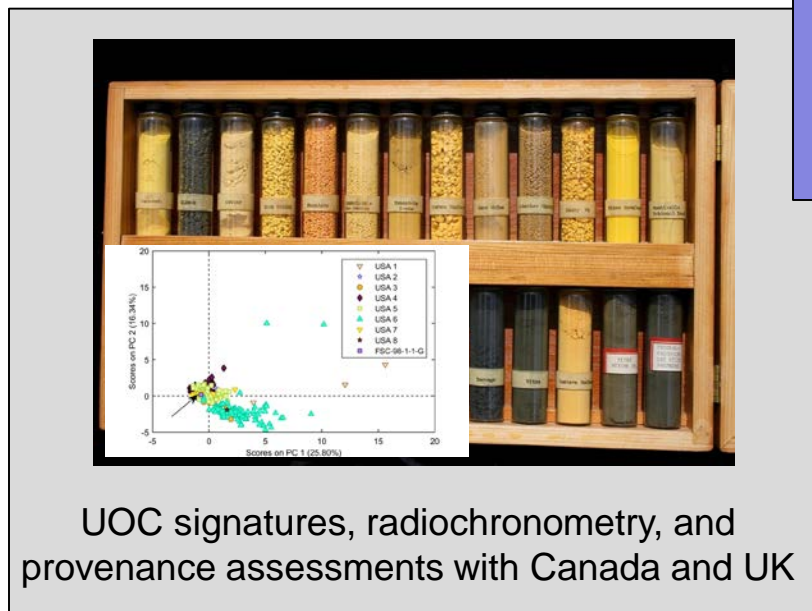
- Bilateral engagement to improve nuclear forensics measurements and build international confidence in data defensibility
 - Targeted R&D
 - Intercomparison exercises

Journal of Radioanalytical and Nuclear Chemistry
<https://doi.org/10.1007/s10967-018-6032-9>

CEA, NIST, LLNL

New determination of the ^{229}Th half-life

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Pa chemistry for radiochronometry and stable oxygen signatures with Israel

C-DHTZ-HM

Multilateral engagement

- International Atomic Energy Agency
 - Instructors for training
 - Subject matter experts for developing guidance
- Global Initiative to Combat Terrorism (GICNT)
 - SMEs for table-top exercises
 - Ensuring guidance for policy makers is technically accurate
- Nuclear Forensics International Technical Working Group (ITWG)
 - Technical exchange, professional development, and interface for scientists and law enforcement
 - International collaborative material analysis exercises (CMX) and NNFL exercises (Galaxy Serpent)





Summary

- Nuclear forensics has emerged as a unique subdiscipline of nuclear science
 - Provides defensible technical data and investigative leads to support criminal investigations, evidentiary needs, and provenance assessments
- Important new application for advancing nuclear science through targeted R&D
 - Material characterization, modeling, and comparative data analysis
 - Building an understanding of signatures as they relate to material provenance and device heritage assessments

Excellent opportunities for young scientists and engineers interested in operational and R&D nuclear science programs!

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